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LABORATORY MANUAL
OF
PHYSICAL CHEMISTRY

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LABORATORY MANUAL
OF
PHYSICAL CHEMISTRY

BY
ALBERT W. DAVISON
Professor of Chemical Engineering
AND
HENRY S. van KLOOSTER
Professor of Physical Chemistry,
Rensselaer Polytechnic Institute

NEW YORK
JOHN WILEY & SONS, Inc.
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PREFACE

In presenting this manual of laboratory exercises in physical chemistry, the authors have been prompted by a threefold object: first, to facilitate the task of the instructor in handling large classes of students beginning a course of practical physical chemistry; second, to assist the student in recording his observations in a clear and comprehensive manner; and third, to present such quantitative exercises as will be particularly desirable in the training of students of engineering.

In view of the fact that there are, as a rule, available for laboratory work not more than two and one-half hours at a time, it has been found desirable to have the different pieces of apparatus set up beforehand by the instructor. To this end, each experiment is preceded by an exact list of the apparatus and chemicals needed. It is believed that this will materially aid the instructor in assembling the necessary equipment. At the Rensselaer Polytechnic Institute, the laboratory course immediately follows the completion of the theoretical course in physical chemistry, and students are thus prepared to take up the study of any experiment herein listed. It is therefore found practical to prepare the equipment for one or two units of each exercise before the laboratory course starts, and to shift the student successively from one experiment to another. In this way, an excessive amount of preparatory work is avoided.

Many of the experiments have been in use in their present form at this Institute, and elsewhere, for a number of years, and have been found to be thoroughly satisfactory.

Directions have been made as concise as possible. Descriptive matter pertaining to thermostats, polarimeters, and other physical measuring instruments has purposely been omitted, since these are adequately described elsewhere. For these detailed descriptions, students are referred to the many excellent laboratory manuals quoted, a number of which may well be kept on the laboratory book-shelves. For theoretical discussions, and formulæ, reference must be had to the textbooks in current usage.

Some of the exercises given are not described in the current manuals, nor indeed in the literature. It is hoped that these contributions may be found useful elsewhere. The number of experiments is limited, but they have been carefully chosen with the aim of covering the whole field of elementary physical chemistry in a *quantitative* manner, so that the student will be enabled to perform *all* of the experiments presented, without omissions, in the time usually allotted to a laboratory course of this character. With the exception of two or three experiments, the majority of these exercises can be performed with the usual apparatus to be found in any chemical laboratory.

Blank sheets and cross-section paper have been introduced in order to make the manual a student note-book as well as a laboratory guide.

The authors take this opportunity to express their appreciation for the kindly criticisms and the many suggestions received from Dr. F. H. Getman, Hillside Laboratory, Stamford, Conn.; Dr. Edward Mack, Jr., Ohio State University; Dr. Harry B. Weiser, The Rice Institute; and Dr. J. Hunt Wilson, Lafayette College. Special recognition should be given to Dr. A. T. Lincoln, of Carleton College, under whose direction the laboratory course in physical chemistry was first established at this Institute. Thanks are due to Eimer & Amend, New York, to the Leeds & Northrup Company, Philadelphia, to Dr. A. F. Holleman and to the publishers for permission to use certain cuts.

Suggestions and criticisms will be appreciated.

THE AUTHORS.

TROY, NEW YORK,
March, 1922.

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Lincoln:	Textbook of Physical Chemistry, rev. Ed., Heath & Co., New York (1920).
Millard:	Physical Chemistry for Colleges, McGraw-Hill Book Co., New York (1921).
Morgan:	Elements of Physical Chemistry, 4th Ed., Wiley & Sons, New York (1908)
Senter:	Outlines of Physical Chemistry, 4th Ed., Van Nostrand Co., New York (1914).
Walker:	Introduction to Physical Chemistry, 9th Ed., Macmillan Co., New York (1922).
Washburn:	Principles of Physical Chemistry, McGraw-Hill Book Co., New York (1915).

B. *Laboratory Manuals:*

Biltz (Jones-King):	Practical Methods of Determining Molecular Weights, Chemical Publishing Co., Easton, Pa. (1899).
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Gray:	Manual of Practical Physical Chemistry, Macmillan Co., London (1914).
Pring:	Laboratory Exercises in Physical Chemistry, University Press, Manchester (1911).
Roth (Cameron):	Exercises in Physical Chemistry, Van Nostrand Co., New York (1920).
Traube (Hardin):	Physico-Chemical Methods Blakiston, Philadelphia (1898).

C. *Special Texts:*

Allmand:	The Principles of Applied Electrochemistry, Longmans, Green and Co., London (1912).
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Bancroft:	Applied Colloid Chemistry, McGraw-Hill Book Co., New (1921).
Dennis:	Gas Analysis, Macmillan Co., New York (1913)
Findlay:	The Phase Rule, 3d Ed., Longmans, Green and Co., London (1911).
Hatschek:	Laboratory Manual of Elementary Colloid Chemistry, Blakiston, Philadelphia (1920).
Holleman (Walker):	A Textbook of Organic Chemistry, 5th Ed., Wiley and Sons, New York (1920).
van Klooster:	Lecture Demonstrations in Physical Chemistry, The Chemical Publishing Co., Easton, Pa. (1919).
Leach-Winton:	Food Inspection and Analysis, 4th Ed., Wiley and Sons, New York (1920).
Leblanc (Whitney, Brown):	A Textbook of Electrochemistry, Macmillan Co., New York (1907).
Lewis:	A System of Physical Chemistry in 3 volumes, 3d Ed., Longmans, Green and Co., London (1920).
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Note. Page numbers cited in the text refer to the page, in the edition listed above, upon which the subject matter under consideration is treated.

LABORATORY MANUAL OF PHYSICAL CHEMISTRY

EXPERIMENT 1A

DETERMINATION OF VAPOR DENSITY

Object:

To determine approximate molecular weights by the vapor density method, using Victor Meyer's apparatus.

References:

Textbooks:

Bigelow, XII, Vapor Densities, p. 165
Getman, II, Gases, p. 35
Jones, II, Gases, p. 60
Lincoln, IV, Determ'n of Mol. & Symb. Wts., p. 32
Millard, II, Props. of Subs. in the Gaseous State, p. 42
Morgan, II, The Gaseous State, p. 18
Senter, II, Gases, p. 37
Walker, XIX, Methods of Mol. Wt. Determ'n, p. 194

Laboratory Manuals:

Biltz (Jones-King), Gas Displacement Method, p. 6
Briggs, II, Vapor Density, p. 8
Ewell, I, Gases, p. 93
Findlay, III, Dens. of Liquids & Gases, p. 49
Firth, II, Dens. of Gases, Liquids & Vapors, p. 8
Getman, II, Volume & Density, p. 30
Gray, Ex. 3, Det. of Vapor Density, p. 39
Roth (Cameron), II, Determination of Density, p. 23
Traube (Hardin), Density, p. 38

Special Texts:

Holleman (Walker), Textbook of Organic Chemistry, Par. II, Determ'n of Mol. Wt., p. 60

van Klooster, Lecture Dem., IV, V. P. & Det. of Mol. Wt., p. 35

Apparatus and Chemicals required:

2 iron stands
2 clamps
2 iron rings
1 wire gauze
1 Bunsen burner+rubber tubing
1 Victor Meyer outer jacket
1 Victor Meyer inner tube suspended in glass jacket
1 graduated (0–100 c.c.) measuring tube (nitrometer tube with double bored stopcock)
1 plain leveling tube+rubber tubing
1 small glass container with capillary stem
1 contrivance to close inner tube and to hold glass container (see Fig. 2)
1 thermometer (0–100°)
1 small Erlenmeyer flask
1 test tube
Sample bottles with aceton, benzene, ether, etc.
Sand
Pumice stone

The whole arrangement is readily understood from Fig. 1. The inner Victor Meyer tube (about 8 mm. in diameter and about 75 cm. long) is enlarged at the bottom so as to hold about 200 c.c. Through a piece of gas-tight rubber tubing it is connected with the measuring tube. The latter is furnished with a two-way stopcock allowing communication with the atmosphere or with the Victor Meyer tube. By raising or lowering the leveling tube containing water the pressure inside the measuring tube can be brought to that of the atmosphere. The outer jacket in which the inner tube is suspended contains water and

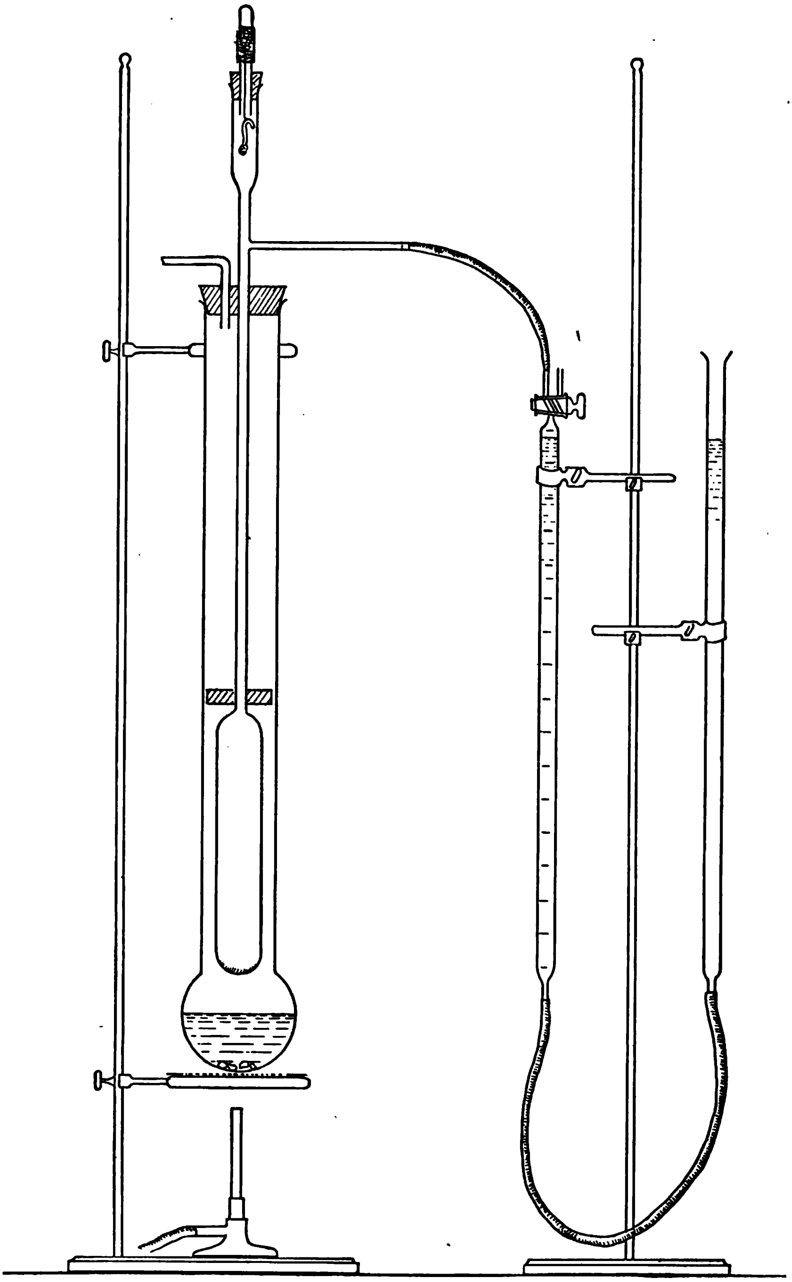

FIG. 1.

[illegible] regular boiling. In order [illegible] ng the jacket, a split cork [illegible] former.

[illegible]

[illegible] contain a little sand) by [illegible] e extending to the bottom [illegible] air through.

[illegible] ube in a central position [illegible] nd see that the bottom is [illegible] of the water in the bulb.

[illegible] bulb with capillary stem [illegible] of liquid) drawn from a [illegible] nd then fill it with the [illegible]

[illegible] some of the liquid into a [illegible] n a small Erlenmeyer flask [illegible] stem of the container, the [illegible] warmed in a flame, in the [illegible] cooling and warming the [illegible] drawn in to cause a weight

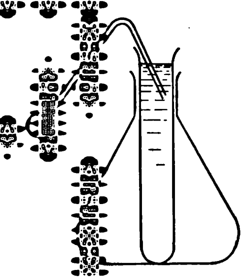

FIG. 3.

[illegible] upper end of the stem over [illegible] again weigh the bulb.

[illegible] iron wire fastened to the [illegible] ctor Meyer tube (Fig. 2), [illegible] uring tube and apply heat [illegible]

When the air within the inner tube has reached the temperature of the steam jacket and is under atmospheric pressure (which is ascertained by connecting the measuring tube alternately to air and to the inner tube and noting whether there is any change of level) turn the stopcock so as to connect the measuring tube with the Victor Mever tube and read the bottom of the meniscus.

Break the stem of the container by a short pull, taking care not to remove the stopper from the inner tube during the operation.

The liquid slowly vaporizes, expelling the air into the measuring tube. Lower the leveling tube to keep the system approximately under atmospheric pressure. When maximum displacement of air has taken place bring the system exactly to atmospheric pressure by adjusting the leveling tube and turn the stopcock through 90° thereby shutting off the air in the measuring tube from the vapor in the Victor Meyer tube. At the same time break the rubber connection between both tubes and remove the flame.

Read the volume of the air in the measuring tube, ascertain the temperature of the water, and the barometric pressure.

Observations and Measurements:

Liquid used, taken from bottle labeled.......
Weight of glass bulb...................... — g.
Weight of bulb+liquid.................... — g.
Same after sealing bulb. .... — g.

Weight of liquid.......................... — g.

Volume reading at start................... — c.c.
Same at end............................... — c.c.

Volume of air collected.................... — c.c.

Temperature of air collected................ — °
Vapor pressure of water at this temperature — mm.[1]
Barometric pressure........................ — mm.
Same corrected.......... .... — mm.
Corrected pressure of displaced air........... — mm.

Calculations:

From the weight of the liquid, the volume, temperature and pressure of the expelled air calculate:

1. The specific gravity of the vapor with respect to air.
2. The density of the vapor under standard conditions.
3. The molecular weight of the compound.

---

[1] See Table I, Appendix.

EXPERIMENT 1B

DETERMINATION OF VAPOR DENSITY

(ALTERNATE METHOD)

Object:

To determine vapor densities using Weiser's modified Victor Meyer apparatus.[1]

References:

See p. 1. Also: Weiser, Jour. Phys. Chem., 20, p. 532 (1916).

Apparatus and Chemicals required:

2 iron stands
1 clamp
3 iron rings
1 wire gauze
1 Bunsen burner+rubber tubing
1 outer jacket for Weiser apparatus
1 Weiser apparatus with cork to fit outer jacket
1 eudiometer tube (50 c.c.)
1 glass or porcelain dish to serve as pneumatic trough
1 thermometer (0–100°)
3 small glass stoppered bottles, capacity 0.2–0.5 c.c.
Sample bottles with benzene, chloroform, ether, etc.
Pumice stone

The assembled apparatus is shown in Fig. 4. The inner vaporization tube is securely held in place within the outer

---

[1] **Obtainable from Eimer and Amend. Also from the Central Scientific Company.**

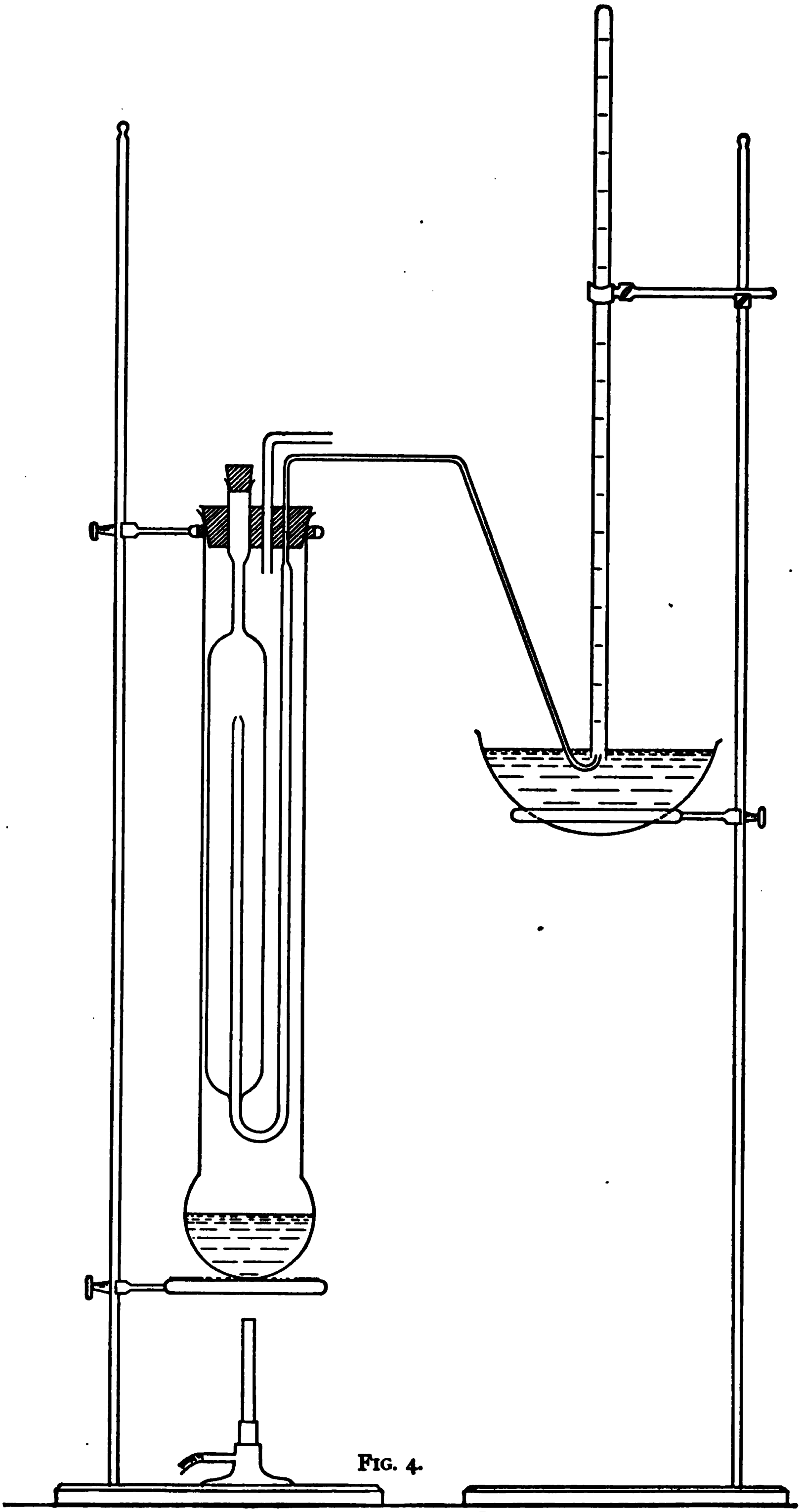

FIG. 4.

jacket by means of a cork split vertically with holes to fit the inlet- and exit-tube of the apparatus and the bent glass tube for the escape of the steam. The two portions of the cork are fastened firmly together by means of a wire around the top.

Method of Procedure:

Fill the bulb of the jacket two-thirds full of water and support the jacket on a wire gauze in a quiet place free from air currents.

Place the inner tube in position and fasten it securely by means of the cork. Fill the dish which serves as pneumatic trough with water and let the end of the capillary delivery tube dip beneath the surface of the water. Fill the graduated eudiometer tube with water, invert it, and clamp it near the delivery tube in such a way that it can be quickly moved over the tip.

Heat the jacket with a low flame and as soon as the water begins to boil, adjust the height of the flame in such a manner that steam just escapes from the bent glass tube.

Weigh about 0.1 gram of the assigned liquid into the small glass stoppered vial and stopper tightly.

Close the vaporization tube by a rubber stopper and continue to boil the water in the jacket. When no more air bubbles are seen to escape from the capillary delivery tube, remove the stopper and place the eudiometer over the tip of the delivery tube.

Loosen up the stopper of the little vial so that it will not stick later on, drop it into the vaporization tube with the left hand and immediately close the tube with the stopper held in the right hand, being careful to secure an air-tight joint.

The sample vaporizes and drives air into the measuring tube.

When air bubbles no longer come over into the eudiometer tube, remove the rubber stopper again, turn off the gas and transfer the stand carrying the eudiometer and pneumatic trough to a place where the temperature of the gas collected, may attain room temperature.

Record the volume of gas, the height of the meniscus of the water inside the eudiometer above the level of the water in the trough, the temperature of the surrounding air and the barometric pressure.

Observations and Measurements:

Liquid used taken from bottle labeled........
Weight of glass stoppered vial+liquid........ — g.
Weight of vial empty...................... — g.

Weight of liquid used...................... — g.

Volume of air collected .... — c.c.
Height of water meniscus .... — cm.
Room temperature......................... —° C.
Barometric pressure....................... — mm.
Same corrected .... — mm.
Vapor pressure of water at room temperature.[1]
Pressure of air collected.. .... — mm.

Calculations:

From the weight of the liquid, the volume, the temperature and the pressure of the air collected calculate:

1. The specific gravity of the vapor with respect to air.
2. The density of the vapor under standard conditions.
3. The molecular weight of the compound.

[1] Consult Table 1, Appendix.

EXPERIMENT 2

GRAHAM'S EFFUSION LAW; DENSITIES OF GASES

Object:

To apply Graham's effusion law to the determination of gas densities.

References:

Textbooks:

Bigelow, XI, Gases, p. 142
Lincoln, VIII, Kin. Theory of Gases, p. 87
Millard, II, Props. of Subs. in Gaseous State, p. 48
Morgan, II, The Gaseous State, p. 19
Senter, II, Gases, p. 33

Laboratory Manuals:

Ewell, I, Gases, Vapors and Liquids, p. 94
Gray, Ex. 2, Determ'n of Dens. of CO_2, p. 38

Special Texts:

Dennis, Gas Analysis, p. 46

Apparatus and Chemicals required:

1 tall glass cylinder, about 4×24 inches
1 effusion tube, dimensions 1×20 inches
1 two-hole rubber stopper to fit effusion tube
2 glass stopcocks, one straight, one bent at right angle
1 wood clamp, with two bolts
1 lead disk, for weighting tube
1 small platinum foil (a 5 mg. weight)
1 stopwatch
Small bore rubber tubing
1 tank of hydrogen or a hydrogen generator

The apparatus (Schilling's) is arranged as shown in Fig. 5. The effusion tube, held tightly at its upper end by means of a wood clamp, weighted by a lead disk is submerged in the water to a certain depth which must not change in order that the average pressure under which the gases effuse remain the same throughout the whole experiment. The hole in the platinum foil through which the gases effuse must be exceedingly small. It is punched by means of a very fine needle and if necessary can be hammered down until a very small opening remains. The foil is cemented to the end of stopcock *A* by means of china cement or universal wax (a mixture of one part of venetian turpentine and four parts of beeswax).

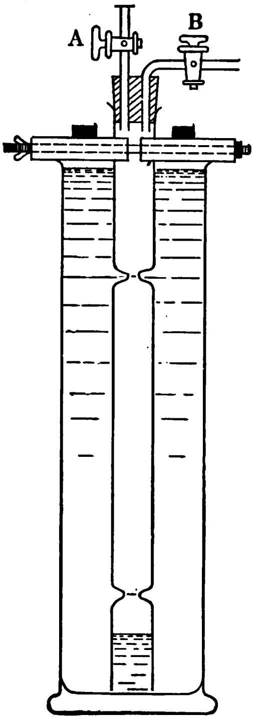

FIG. 5.

Method of Procedure:

Close stopcock *A*, open *B* and fill the effusion tube with air, either by blowing it in from the laboratory air lines or by lifting the tube out of the water.

Close *B*, replace the tube and see that the water in the outer cylinder rises to within an inch of the top.

See that the water in the apparatus is saturated each time with the gas under observation and that it has the temperature of the room.

With stopwatch in hand, open stopcock *A* and allow the air to pass through the opening in the foil. Start the watch when the rising water passes the mark on the lower constriction of the effusion tube and stop it when the water passes the mark on the upper constriction.

Fill the tube again with air and repeat the observation and continue until at least three determinations have been made which check to two-fifths of a second.

Open both stopcocks and pass hydrogen through the apparatus until all the air has been replaced by hydrogen, then close *A* and fill the tube with hydrogen, raising it if necessary to secure the entry of a sufficient amount of gas.

Close *B*, open *A* and record the time required for this gas to effuse through the opening. Repeat the operation until at least three good checks have been obtained.

Replace the residual hydrogen with illuminating gas from the laboratory fuel lines, performing the operation under the hood to prevent the escape of toxic gases into the room.

Operate as before and obtain at least three determinations which check to two-fifths of a second.

Record of Observations:

Gas used . . . .
Effusion time . . . ., ..., ..., ...

Gas used . . . .
Effusion time . . . ., ..., ..., ...

Gas used . . . .
Effusion time . . . ., ..., ..., ...

Gas used . . . .
Effusion time . . . ., ..., ..., ...

Calculation of Results:

1. By means of Graham's effusion law, calculate the specific gravity (air = 1) of each of the gases used.
2. From the specific gravities, calculate the density of each gas.
3. Calculate the molecular weight (for illuminating gas the "apparent" molecular weight).

EXPERIMENT 3

VAPOR PRESSURE OF LIQUIDS

Object:

To determine the vapor pressure of liquids at different temperatures by means of the isotensiscope.[1]

References:

Textbooks:

Bigelow, XXI, Vapor Pressure, p. 274
Getman, III, Liquids, p. 75
Jones, III, Liquids, p. 99
Lincoln, XVI, Soln's of Liquids in Liquids, II, p. 168
Millard, II, Prop's of Subs. in Gaseous State, p. 71
Morgan, III, The Liquid State, p. 64
Senter, V, Dilute Solutions, p. 114
Walker, VIII, Vaporiz'n & Condensation, p. 72
Washburn, IV, Liquid-Gas Systems, p. 53

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Smith and Menzies, Jour. Am. Chem. Soc. 32, p. 1412 (1910).
Derby and Yngve, Ibid. 38, p. 1439 (1916).
Edgar and Swan, Ibid. 44, p. 570 (1922).

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[1] As the instrument indicates the equality of tensions it appears that isotensiscope is a better name than the usual name of isoteniscope, although the latter is probably grammatically correct.

Apparatus and Chemicals required:

1 iron stand
3 clamps
1 condenser
1 stirrer, driven by a small motor
1 2-liter beaker[1]
1 electric hot plate
1 thermometer (0–110°)
1 barometer tube and 1 open mercury manometer, attached to the same meter stick (inner diameter of both tubes 3 mm., length 90 cm.)
1 4-gallon bottle
1 water pump
1 isotensiscope with tight-fitting ground-glass joint
1 two-way stopcock and 1 ordinary stopcock
Sample bottles containing liquids to be tested.

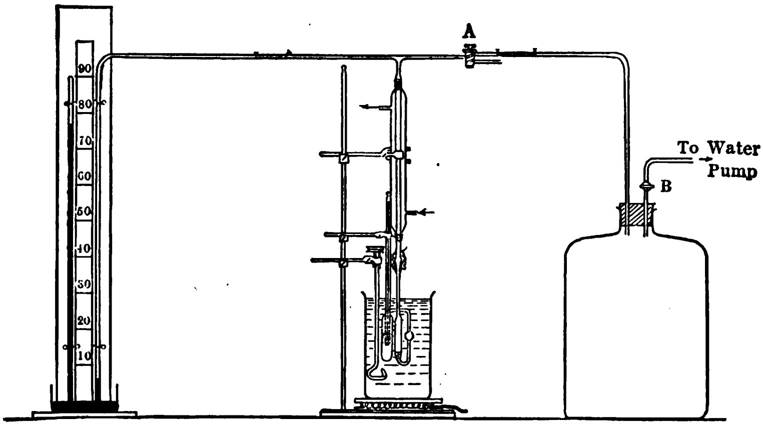


FIG. 6.

The arrangement is readily understood from Fig. 6. The isotensiscope consists of a cylindrical bulb of about 25-c.c. capacity sealed to a 10-c.c. pipette bent as shown and connected to a reflux condenser by means of a ground-glass joint. The condenser communicates on the left with the open manometer and on the right, through a two-way stopcock A, either with the air or with a large 4-gallon bottle which serves as a pressure regulator. By means of a bent tube provided with a stopcock B, connected to a water pump the system can be evacuated.

---

[1] Instead of a beaker a large size unsilvered Dewar vacuum tube with an electrical heating arrangement inside may be used.

Method of Procedure:

Assemble the apparatus as shown in the figure, connecting the empty isotensiscope, cleaned and dry, to the condenser, and apply suction.

Shut B, see that the pressure is maintained and that no leaks develop. The pressure of the system is represented by the difference in height of the mercury in both tubes (accurate to 1 mm. mercury). The barometer reading is compared with that of the standard laboratory barometer and a correction applied if necessary.

When found to be in working order, the pressure is brought back to atmospheric by carefully and slowly opening A to air.

Disconnect the isotensiscope and fill the cylindrical bulb two-thirds full of the liquid under observation, using diminished pressure to force the liquid from the pipette into the bulb. Make a tight joint with the condenser.

Fill the beaker with water (or glycerol, for temperatures above 100°), insert the thermometer placing the bulb next to the middle of the container, and stir the liquid *vigorously* by means of the motor-driven stirrer.

When the temperature approximates the boiling point of the liquid some of it distills over and condenses in the bent part of the isotensiscope.[1]

The heat is then turned off and the substance allowed to cool. The bubbles continue to flow through the liquid until the liquid cools to the boiling-point temperature (at atmospheric pressure), when bubbling ceases, and the liquid is on the point of being sucked back into the small bulb on the right.

---

[1] This preliminary boiling is continued for some time to remove all the air and insure a uniform temperature throughout the liquid. For mixtures to which the operation can also be applied the boiling should be for a short period only as prolonged boiling will change the composition considerably.

As soon as the liquid reaches the bottom of the small bulb the pressure and the temperature are read.

The two-way stopcock *A* is then immediately opened to the pressure reservoir, stopcock *B* is opened and suction applied. The mercury in the open manometer is allowed to rise about 5 cm. and the stopcock *B* closed. The liquid boils again, bubbles flow through the bend until on further cooling the flow of bubbles stops and the liquid again sucks back as before. Temperature and pressure are then read and noted. This cycle of operations, known as the method of Ramsay and Young, is repeated until the liquid has cooled down sufficiently (to about 20° C.).

Finally the stopcock *A* is slowly opened to air and the system again brought under atmospheric pressure. *B* is then opened and the water pump shut off.

Stop the motor, disconnect the isotensiscope and remove the liquid from the instrument.

Observations and Measurements:

Liquid used ....
Barometer reading .... — mm.
Same corrected .... — mm.

Readings:

Temperature	Pressure	Same corrected
......	......	......
......	......	......
......	......	......
......	......	......
......	......	......
......	......	......
......	......	......
......	......	......
......	......	......
......	......	......
......	......	......
......	......	......

Plotting the Results:

Plot the results on coordinate paper, the pressures as ordinates against the temperatures as abscissae, on a sufficiently large scale and connect the points thus obtained by a smooth curve.

EXPERIMENT 4

SURFACE TENSION AND ASSOCIATION FACTOR OF LIQUIDS

Object:

To determine the surface tension and the molecular surface energy of an assigned liquid and to calculate its association factor.

References:

Textbooks:

Bigelow, XIII, Liquids, p. 180
Getman, III, Liquids, p. 86
Jones, III, Liquids, p. 145
Lincoln, XI, Phys. Props. of Liquids, p. 115
Millard, III, Props. of Subs. in the Liq. State, p. 67
Morgan, III, The Liquid State, p. 78
Senter, V, Dilute Solutions, p. 125
Walker, XIX, Meth. of Mol. Wt. Determ'n, p. 206
Washburn, III, The Liq. State of Aggregation, p. 43

Laboratory Manuals:

Ewell, I, Gases, Vapors & Liquids, p. 177
Findlay, V, Viscosity & Surf. Tens., p. 88
Firth, III, Determ'n of Vis. & Surf. Tens., p. 18
Getman, IV, Vis. & Surf. Tens., p. 52
Gray, Ex. 15, Surface Energy, p. 65
Traube (Hardin), III, Capillarity, p. 45

Apparatus and Chemicals required:

1 iron stand
1 iron ring
1 clamp
1 tall 2-liter beaker
1 large test tube 35 cm. long, diameter 4 cm.
2 capillary glass tubes with uniform bore, 35 cm. long, diameter of bore about 0.3–0.5 mm.
1 stirrer for beaker
2 small scales graduated in mm.
1 thermometer (0–100°)
1 rubber tube, 5 mm. bore, 50 cm. long
1 Bunsen burner+rubber tubing
1 wire gauze
1 bottle containing dry purified mercury
1 bottle containing cleaning solution
1 bottle containing distilled water
Sample bottles containing other liquids to be tested

The apparatus as used in this experiment is illustrated in Fig. 7.

The liquid under observation is placed in the large test tube. The latter is supported vertically in the beaker which serves as a water bath. The capillary tubes passing through a three-holed cork are mounted vertically with their lower ends immersed in the liquid in the test tube. The graduated scales are fastened to the lower ends of the capillary tubes by means of tinned wire.

Method of Procedure:

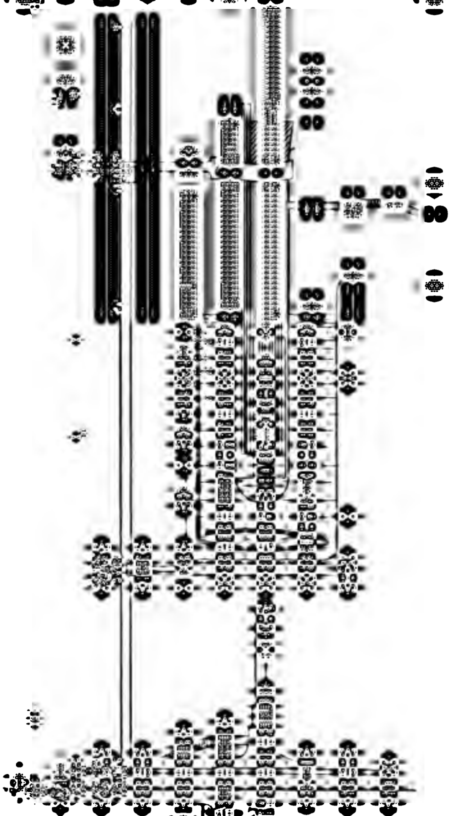
Mark the capillary tubes in such a manner that they can be easily identified during the experiment.

Introduce a column of mercury about 10 cm. long into each of the tubes and measure the length accurately.

Weigh the mercury and calculate the diameter of the tubes.

Clean the bores of both tubes thoroughly with hot cleaning solution, wash out with water and finally rinse with distilled water. All other apparatus coming in contact with the liquid [illegible] aned and kept clean during [illegible]

[illegible] scales in a vertical position and fix them there by adjustment of the cork through which they are suspended.



[illegible]

Place sufficient freshly distilled water (or other assigned liquid)[1] inside the test tube to bring the level on the scales.

Fill the beaker with tap water, and, using the stirrer, bring the temperature to such a value that the liquid inside the test tube is at 20°.

Blow gently into the rubber tube connected to the test tube, in order to force the liquid up into the capillaries to a height greater than that of equilibrium; this is to "wet" the tubes, and to insure that the liquid will rise to equilibrium height. Avoid [illegible] portions of the capillaries.

[illegible] the liquid rises three times, [illegible]tly blowing into the rubber [illegible]

---

[1] [illegible] under observation, for the reasons [illegible] liquids, and that it is not necessary [illegible]ervations are to be made on liquids [illegible]illaries by drawing a current of hot [illegible]han to employ the usual procedure [illegible]atter liquids are very likely to contain [illegible]apillaries.

Raise the temperature to 40° and then to 60° and take three readings in each instance.

Observations and Measurements:

	Tube 1	*Tube 2*
Length of column of mercury.......	......	......
Weight of same....................	......	......
Density of mercury at room temp....		
Diameter of the tube..............	......	......
Liquid used		
Capillary rise at 20°.............	......	......
	......	......
	......	......
Average....................	......	......
Capillary rise at 40°.............	......	......
	......	......
	......	......
Average....................	......	......
Capillary rise at 60°	......	......
	......	......
	......	......
Average....................	......	......

Calculations:

1. Calculate the surface tension and the molecular surface energy for each temperature.
2. Determine the temperature coefficient between 20° and 40° and between 40° and 60°.
3. From the two values of K thus obtained, calculate the association factor between the limits 20°–40° and 40°–60°.

EXPERIMENT 5

REFRACTIVE INDEX, DENSITY AND MOLECULAR REFRACTIVITY OF LIQUIDS

Object:

To determine the index of refraction and the density of a given liquid at the same temperature and to find its specific and molecular refractivity.

References:

Textbooks:

Bigelow, XIV, Index of Refraction, p. 190
Getman, V, Phys. Props. & Mol. Const'n, p. 124
Jones, III, Liquids, p. 115
Lincoln, XII, Refraction of Light, p. 122
Millard, XI, Phys. Props. & Mol. Structure, p. 316
Morgan, III, The Liquid State, p. 77
Senter, III, Liquids, p. 63
Walker, XV, Rel'n of Phys. Props. to Comp'n & Constit'n, p. 150
Washburn, VIII, Rel'n Phys. Props. & Chem. Constit'n, p. 80

Laboratory Manuals:

Ewell, V, Light, p. 216
Findlay, VI, Optical Measurements, p. 97
Firth, VIII, Refractivity Measurements, p. 46
Getman, VIII, Optical Measurements, p. 138
Gray, Ex. 26, Det. of Mol. Refractive Power, p. 107
Roth (Cameron), V, Det. of Opt. Constants, p. 70
Traube (Hardin), XVII, Index of Refraction, p. 168

Special Texts:

Winton, Food analysis, VII, Fats & Oils, p. 149
Woodman, Food analysis, I, Gen. Methods, p. 7

Apparatus and Chemicals required:

1 refractometer
1 thermostat, preferably at 20° C.
1 pyknometer and pipette with capillary stem (Fig. 8)
1 bottle containing distilled water
Sample bottles containing different organic liquids
Silk paper, to clean prisms of refractometer

Fig. 9 represents the Abbe refractometer, which is widely used in this country and is most satisfactory for general laboratory practice, as it is easy to handle and is so constructed that it can be used with daylight and gives values corresponding to the "D" line. For accurate setting it is necessary, however, to use sodium light (see p. 99). It consists of two prisms of flint glass with a refractive index of 1.75 which can be separated so as to allow the introduction of a few drops of liquid. The prisms can be rotated by means of a movable arm *M* which carries the reading glass *R*. The position of the border line of total reflection is observed through the fixed telescope *T* and by turning the movable arm it is made to coincide with the intersection of the cross hairs in the telescope. The arc *A* is graduated so as to read directly refractive indices (between 1.3 and 1.7) to the third decimal place. The fourth decimal is estimated with an accuracy of 2 units by means of the lens *R*. The compensator *C* composed of two similar Amici prisms, rotated simultaneously in opposite directions by the milled head *H*, is used to produce a dispersion (*C*—*F*) equal but opposite to that of the liquid and permits the border line to be changed from a colored band to a sharp colorless line. By taking the reading on the divided drum *D* and using the table supplied with the instrument the dispersion may also be found. A heating arrangement furnished by the makers, or else a large thermo-

[illegible] lation of water at a given [illegible] the casing of the prisms.

[illegible]

[illegible] ent on a table near the [illegible] direct sunlight, and provide [illegible] temperature bath, making [illegible] circulation of the water [illegible] a slow but steady stream. [illegible] release the lower prism [illegible] position shown in Fig. 9. [illegible] he correctness of the ad- [illegible] ctometer, place a drop of [illegible] glass surface, close the [illegible] em by means of the latch *L*, [illegible] turning the movable arm

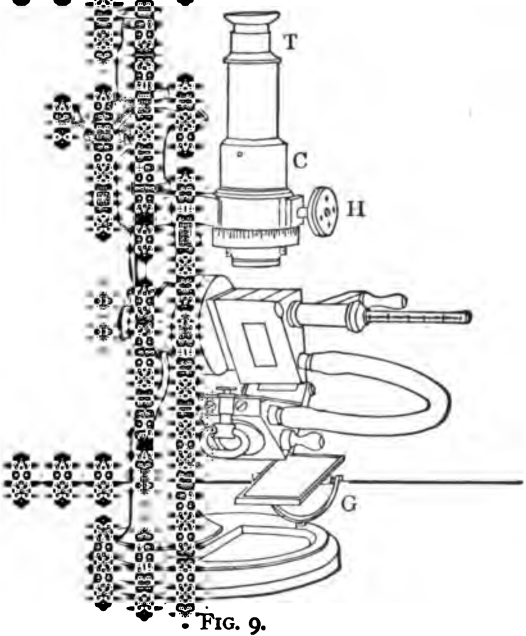


Fig. 9.

[illegible] way that it reflects light [illegible] appears in the field.

Turn the screw head *H* and thereby adjust the compensator so that the colored band due to dispersion disappears and a sharp boundary line is obtained.

Finally rotate the arm *M* until the line coincides with the intersection of the cross hairs and take the reading to the fourth decimal place. Also read the temperature.

Repeat the readings at least three times and take the mean, comparing the result with the data given for water on Table 3 of the Appendix.

Open the latch, drop the lower prism, clean the polished glass surfaces with a little alcohol and silk paper (do not use filter paper) and operate on the liquid(s) assigned for investigation in exactly the same manner as outlined above.

In order to be able to calculate the molecular refractivity it is necessary to determine the density of the liquid at the temperature for which the readings were taken. Any of the well-known types of pyknometers may be used. A special form of pyknometer which the authors prefer is represented in Fig. 8.[1] It has the advantage that it is not fragile, can be operated equally well at temperatures above and below that of the room, is readily cleaned and can be made in a very short time by any-one of moderate skill in glass blowing. In case the pyknometer has been calibrated at different temperatures, only one observation of the liquid at the desired temperature, and one weighing is required to find the density for that temperature.

The density is then given by the formula:

$$d_{4^\circ}^{t^\circ} = \frac{W'D}{W} - \frac{0.0012\,(W' - W)}{W}$$

in which W' is the weight of liquid at t°, W the weight of water at t°, and D the density of water at t° (see Table 4 of the Appendix). The small fraction to be subtracted is a correction for the buoyancy of the air.

The stem of the pipette (see Fig. 8) is of such length and width that it passes though the narrow capillary tube connecting the upper and lower reservoir of the pyknometer and reaches down to the bottom of the lower bulb.

---

[1] Bingham, van Klooster and Kleinspehn, Jour. Phys. Chem., **24**, p. 6 (1920).

To fill the pyknometer, connect the pipette to the suction pump and let it hang free from a ring stand. Fill the top reservoir of the clean, dry pyknometer with the liquid under investigation, then pass the capillary stem of the pipette through the capillary connection of the pyknometer, and withdraw the air from the bulb, by suction. Add enough liquid to fill the upper reservoir to a height of a few mm., place the instrument in the thermostat and after thermal equilibrium has been reached, withdraw just enough liquid by means of the pipette to bring the level in the capillary connection to the mark. Dry the pyknometer and weigh it.[1] Empty the pyknometer by lowering the stem of the pipette to the bottom and suck the liquid out using a water pump as before. Wash with alcohol, then ether and finally suck a current of dry air through the instrument.

Find the weight of the pyknometer, first empty and then filled with water of the same temperature (or, if the time is limited take these values from a table compiled for the pyknometer).

Observations and Measurements:

A. *Refractometer Measurements:*

Water at ....° C. Refractive index: ......
......
......
......

Average value:
Value given:

Liquid used: ....
Temperature ....° C. Refractive index: ......
......
......
......

Average value:

---

[1] For volatile liquids, close the upper reservoir with a cork; for *very* volatile liquids like ether a small bulb should be blown in the stem just above the mark.

B. *Pyknometer Measurements:*

Weight pyknometer empty: .... g.
Same filled with water at ....° C. .... g.
Same filled with liquid at ....° C. .... g.

Calculations:

1. From the observations under A and B calculate the specific and the molecular refractivity of the liquid under observation, using both the Gladstone-Dale and the Lorenz-Lorentz formulæ, and compare the values obtained with those given in the literature (see the tables of Landolt-Börnstein).

EXPERIMENT 6A

LOWERING OF THE FREEZING POINT

(BECKMANN METHOD)

Object:

A. To determine the approximate molecular weight of a non-dissociating compound in solution.

B. To determine the apparent molecular weight of an electrolyte in solution and to calculate the degree of dissociation of the electrolyte at the concentrations employed.

References:

Textbooks:

Bigelow, XXIII, Vap. Press. of Sol'ns, p. 324
Getman, VIII, Dil. Soln's & Osm. Press., p.
Jones, V, Solutions, p. 228
Lincoln, XXV, Fr.-Pts. & Boil.-Pts. of Soln's, p. 278
Morgan, VI, Solutions, p. 172
Senter, V, Dilute Solutions, p. 120
Walker, XIX, Determ'n of Mol. Wts., p. 204

Laboratory Manuals:

Biltz (Jones-King), Det. of Mol. Wt. by Fr.-Pt. Meth., p. 73
Briggs, VI, Depression of Fr.-Pt., p. 21
Ewell, I, Gases, Vapors & Liquids, p. 35
Findlay, VII, Mol. Wt. of Subs. in Soln., p. 125
Firth, V, Determ'n of Mol. Wts., p. 31
Getman, VI, Melting & Boiling Points, p. 70
Gray, Ex. 11, Lowering of Fr.-Pt., p. 57
Pring, Part I, Phys. Chem. Meas., p. 4
Roth (Cameron), III, Determ'n of Mol. Wts. in Sol'n p. 43
Traube (Hardin), IX, Depression of Fr.-Pts. of Soln's, p. 82

Apparatus and Chemicals required:

1 battery jar for freezing mixture
1 metal cover for same
1 metal pan
1 glass air jacket
1 Beckmann freezing point tube
1 Beckmann thermometer
1 thermometer (from −10 to 100°)
2 stirrers, one small, one large
1 reading glass
1 10-c.c. pipette
1 two-hole cork carrying the Beckmann thermometer and small stirrer for freezing point tube
1 large cork for adapting freezing point tube to air jacket
Ice and salt for freezing mixture and sample tubes with solute (sugar, urea, and salts)

The apparatus is shown in Fig. 10. A mixture of water, crushed ice and salt, sufficient to reach within an inch of the top, is placed in the large glass jar. The air jacket is fixed in this freezing mixture through the central aperture of the cover, and serves to prevent direct contact between the Beckmann freezing point tube and the cooling bath.

Method of Procedure:

Prepare a freezing mixture and maintain its temperature around −5°.

By means of a 10-c.c. pipette introduce sufficient distilled water into the freezing tube to immerse the bulb of the Beckmann thermometer. Make each addition exactly 10 c.c. The mass of water used is determined by noting the temperature and obtaining the weight from Table 4, Appendix. Place the thermometer and stirrer in position in such a manner that the stirrer moves freely, and immerse the tube directly in the freezing mixture. It will be noticed that the water supercools several degrees before freezing begins, and that as soon as ice forms the [illegible] ing the freezing point (heat [illegible] te).

[illegible] rom the cold bath, wipe it [illegible] allowing equilibrium to be [illegible] ained. Stir gently, tapping [illegible] stem of the thermometer [illegible] regular intervals and record [illegible] dings every 30 seconds, esti[illegible] ting thousandths of a degree [illegible] means of a reading glass. [illegible] value obtained when read[illegible] s have become constant is [illegible] en as the freezing point of [illegible] re water. Plot these results [illegible] coordinate paper. Melt the [illegible] and repeat the determina[illegible] n. Successive observations [illegible] ould agree to within 0.002° [illegible] fore proceeding.

[illegible] Introduce about half a gram [illegible] solute, accurately weighed, [illegible] determine the freezing point [illegible] the solution. Inasmuch as [illegible] separation of the solid phase [illegible] e) concentrates the solution, [illegible] s necessary to exercise ex[illegible] me care in having the smallest [illegible] sible amount of ice present [illegible] the time of final temperature [illegible] ding. In case this precau[illegible] n is not observed, results will [illegible] ions, it is best not to immerse [illegible] cold freezing mixture. Should [illegible] order to initiate the crystal[illegible] greater portion of the ice by [illegible] lacing it in the air bath. As [illegible] onds until the equilibrium temperature has been located, and plot these values, taking the maximum temperature on the temperature-time curve.

Add two successive additional weighed portions of about 0.2 gm. of solute, and determine the freezing point of each solution.

Observations and Measurements:

Volume of distilled water used .... — c.c.
Temperature ....°
Density (from Table 4) ....
Weight of water used .... — g.

Thermometer readings at half-minute intervals:

	Water	Solution 1	Solution 2	Solution 3
	......	......	......	......
	......	......	......	......
	......	......	......	......
	......	......	......	......
	......	......	......	......
	......	......	......	......
	......	......	......	......
	......	......	......	......
	......	......	......	......
	......	......	......	......
	......	......	......	......
	......	......	......	......
	......	......	......	......
	......	......	......	......
	......	......	......	......
	......	......	......	......
	......	......	......	......
	......	......	......	......
	......	......	......	......
	......	......	......	......
Solute added				
Weight in g.		......	......	......
Lowering of freezing point		......°	......°	......°

Calculations:

A. Solute is a non-electrolyte:

1. Calculate the molecular weight of the solute for each concentration used. Obtain freezing-point constant from Table 5, Appendix.

B. Solute is an electrolyte:

1. Calculate the apparent molecular weight of the solute for each concentration used.
2. Compute the degree of dissociation for each apparent molecular weight.
3. Plot apparent molecular weight, and degree of dissociation, against concentration, expressed in grams of solute per 100 grams of solvent.

EXPERIMENT 6B

LOWERING OF THE FREEZING POINT BY A NON-ELECTROLYTE

(DEWAR VACUUM TUBE METHOD)

Object:

To determine the molecular weight of *urea* by the freezing-point method, using a vacuum-jacketed Dewar tube.

Journal Articles:

Roloff, Zeits. f. phys. Chemie, 18, p. 572 (1895)
Ponsot, Ann. chim. phys. (7) 10, p. 79 (1897)
Richards, Jour. Am. chem. soc. 25, p. 29 (1903)

Apparatus and Chemicals required:

- 1 vacuum cylinder (suitable dimensions 6 cm. × 20 cm., contents 500 c.c. or 7 cm. × 30 cm., contents 1000 c.c.) or a 1 qt. wide-mouth Thermos flask.
- 1 cork for mouth of same carrying:
    - 1 Beckmann thermometer
    - 1 glass tube with linen filter at lower end
    - 1 short glass tube
    - 1 glass stirrer
- 1 reading glass
- 1 cork to protect tip of Dewar tube
- 1 corrugated cardboard packing filler
- 2 iron stands
- 2 iron rings
- 3 clamps
- 1 50-c.c. pipette
- 1 10-c.c. pipette

1 5-c.c. pipette
2 250-c.c. wide-mouth Erlenmeyer flasks
2 15-c.c weighing bottles, short form
1 one-hole rubber stopper for Erlenmeyer, fitted with piece of glass tubing and short rubber connection
1 graduated (0–100 c.c.) gas measuring tube
1 leveling tube+rubber tubing
1 thermometer (0–100°)
1 small bottle containing about 20 c.c. freshly made saturated urea solution
1 500 c.c. bottle containing a solution of sodium hypobromite freshly prepared by dissolving 100 grams of caustic soda in 250 c.c. of water and adding little by little 25 c.c. of bromine
1 ice shaver or chipper
Ice

The assembled apparatus is shown in Fig. 11. The vacuum flask, with its tip protected by a cork, is wrapped in packing cardboard to prevent injury and held securely on an iron stand by means of two iron rings. The cork fitting into the mouth of the flask is pierced by four holes: the central one carrying the Beckmann thermometer and the three others the stirrer, a short glass tube through which urea is introduced and a longer tube through which samples are withdrawn. This latter tube should be of such length that the pipette used for withdrawing solutions reaches just to the linen filter.[1]

Method of Procedure:

Fill the Dewar vessel about two-thirds full of shaved or finely crushed ice and add sufficient distilled water to permit the stirrer to move freely.

See that the Beckmann thermometer is "set" so that the mercury comes to rest near the top of the scale when the bulb is immersed in ice-water. Place the thermometer in position with its bulb near the center of the mixture of ice and water.

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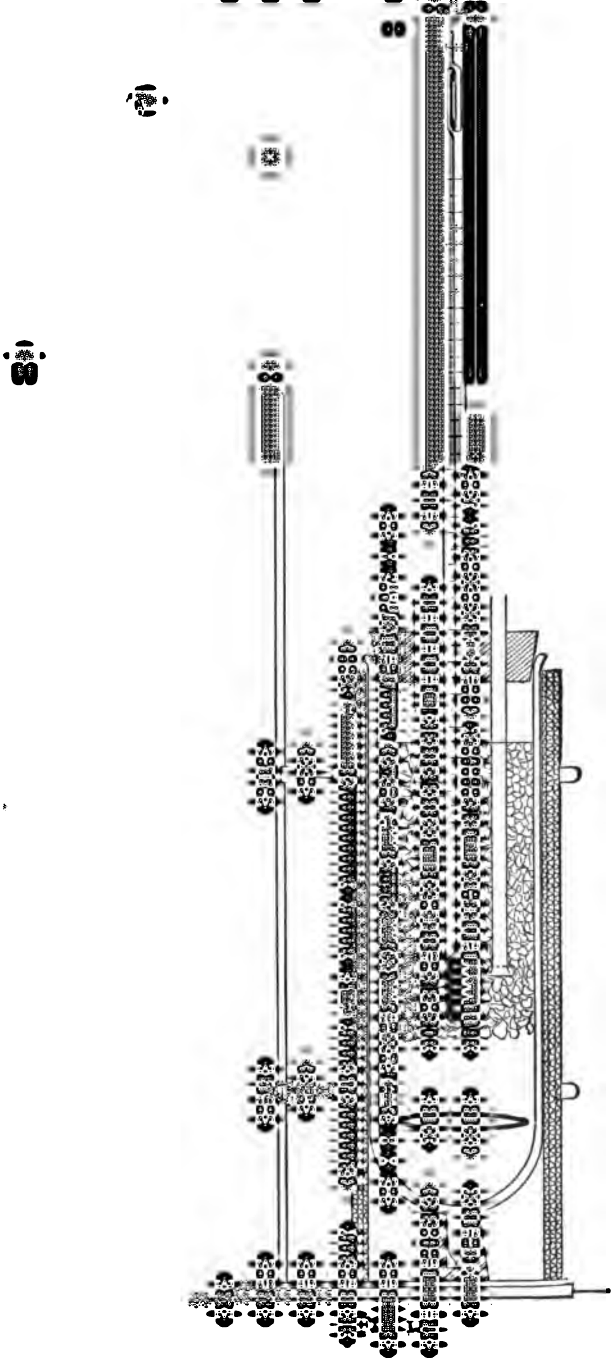
[1] The apparatus as described here has been successfully used in this laboratory for a number of years by Dr. A. T. Lincoln.



Stir regularly but not violently and take temperature readings at half-minute intervals until five successive constant values have been obtained. This constant value is taken as the freezing point of pure water.

By means of a 5-c.c. pipette introduce an amount of urea solution sufficient to lower the freezing point about two-tenths of a degree. Determine the freezing point of this solution by the method outlined above.

Without delay, pipette off through the long glass tube 10 c.c. of solution and reject this; then take two 10-c.c. samples for analysis and run these into the 15-c.c. weighing bottles, placed inside the 250 c.c. Erlenmeyer flasks.

By means of a 50 c.c. pipette put about 50 c.c. of the hypobromite solution in each flask, being careful that the two solutions do not mix

Insert the perforated rubber stopper, place one Erlenmeyer flask in a clamp, and connect it with the gas measuring tube (nitrometer).

Raise the water in the nitrometer to a point near the top, secure atmospheric pressure within the system and take the initial volume reading.

By a gentle swinging, upset the small weighing bottle and allow the two solutions within the Erlenmeyer flask to mix thoroughly. As fast as gas is evolved, lower the leveling tube in order to keep the system under approximate atmospheric pressure.

When the gas evolution is complete, allow the reaction vessel to come to room temperature, bring the system to atmospheric pressure and record the final volume.

Also record the barometric pressure and the room temperature.

In exactly the same manner determine the volume of gas for the sample in the other flask.

Following the method outlined above, 1 c.c. of gas (nitrogen) *under standard conditions* is equivalent to 0.00288 gram of urea.

Add two additional volumes of urea solution to the vacuum flask, so that a further lowering of about 0.2° and 0.4° is produced and in each case determine as before the freezing point and the concentration of urea.

It is advisable that one student carry out the freezing-point determinations and his partner the gas evolution measurements.

Observations and Measurements:

Temperature readings at half-minute intervals:

Ice-water	Solution 1	Solution 2	Solution 3
....	....	....	....
....	....	....	....
....	....	....	....
....	....	....	....
....	....	....	....
....	....	....	....
....	....	....	....
....	....	....	....
....	....	....	....
....	....	....	....
....	....	....	....
....	....	....	....

Nitrometer readings before and after gas evolution:

....	....	....	Sample 1
....	....	....	
....	....	....	
....	....	....	Sample 2
....	....	....	
....	....	....	
....	....	....	Average

Barometric pressure ....mm. Corrected ....mm.
Temperature ....° Vapor pressure of water ....mm.
Corrected gas pressure ....mm.

Calculations:

1. Reduce the three (average) gas volumes to standard conditions and calculate the weight of urea present in each sample. Inasmuch as the solutions are very dilute these weights may be taken as being present in 10 grams of water.

2. Calculate the molecular weight of urea for each set of observations.

3. Plot molecular weight against concentration and connect the points by a straight line. Project this line to the Y-axis; the point of intersection representing the value for infinite dilution is the nearest approach to the theoretical molecular weight.

EXPERIMENT 6C

LOWERING OF THE FREEZING POINT BY AN ELECTROLYTE

(Dewar Vacuum Tube Method)

Object:

To determine the "apparent" molecular weight of an acid in aqueous solution, and to calculate the degree of dissociation of this electrolyte at the concentrations employed.

Apparatus and Chemicals required:

1 wide-mouth vacuum flask with accessories as described in Experiment 6B. Instead of the nitrometer outfit therein mentioned, substitute:
1 iron stand
1 burette holder
1 burette
1 10-c.c. pipette
1 2-c.c. pipette
2 wide mouth 125-c.c. Erlenmeyer flasks
1 small bottle with dilute (1 : 3)HCl or other acid
1 stock bottle with tenth normal alkali
1 phenolphthalein indicator solution

For a sketch of the apparatus see Fig. 11.

Method of Procedure:

Fill the Dewar vessel with ice and water as described on page 44 and determine the freezing point of water in the manner outlined before.

By means of a 2-c.c. pipette introduce a sufficient amount of the acid through the short glass tube, to lower the freezing point about one-tenth of a degree. Stir regularly and take half-minute readings until five successive constant readings are obtained.

Having determined the freezing point of this solution, immediately remove 10 c.c. of the solution through the filter tube, reject this, and then place two 10-c.c. samples for titration in the small Erlenmeyer flasks, labeled 1 and 2.

Titrate these solutions with standard alkali, using phenolphthalein as indicator.

Add three additional successive amounts of acid to the vacuum flask, each giving a further lowering of about one-tenth of a degree, and determine the freezing point and the strength of the solution in each case.

It is advisable that one student carry out the freezing-point determinations, and his partner the titrations.

Observations and Measurements:

Acid used in the experiment ....
Normality of alkali used for titration ....
Temperature readings at half minute intervals:

Ice-water	Solution 1	Solution 2	Solution 3	Solution 4
......	....	....	....	....
......	....	....	....	....
......	....	....	....	....
......	....	....	....	....
......	....	....	....	....
......	....	....	....	....
......	....	....	....	....
......	....	....	....	....
......	....	....	....	....
......	....	....	....	....
......	....	....	....	....
......	....	....	....	....

Number of c.c. of base used in titrating the solutions:

Sample 1	....	....	....	....
Sample 2	....	....	....	....
Average	....	....	....	....
Normality of solution	....	....	....	....
Grams of acid in 100 grams of H_2O	....	....	....	....

Calculations:

1. Assuming, for the dilute solutions used, that the weight of acid found in 10 c.c. of solutions may be taken as present in 10 grams of water, substitute the weights of solute present in 100 grams of water in the table above.

2. By means of the freezing-point equation calculate the apparent molecular weight for each concentration.

3. From the apparent molecular weights calculate the degree of dissociation.

4. Plot the values found for the dissociation against the concentrations as abscissae.

EXPERIMENT 7

MOLECULAR WEIGHT DETERMINATION

(Boiling-Point Method)

Object:

To determine approximate molecular weights by the boiling-point method, using Eykman's apparatus.

References:

Textbooks:

Bigelow, XXIII, Vap. Press. of Soln's, p. 319
Getman, VIII, Dil. Soln's & Osm. Press., p. 246
Jones, V, Solutions, p. 262
Lincoln, XXV, Fr.-Pts. & Boil.-Pts. of Soln's, p. 279
Millard, V, Solutions, p. 125
Morgan, VI, Solutions, p. 167
Senter, V, Dilute Solutions, p. 114
Walker, XIX, Determ'n of Mol. Wts., p. 198

Laboratory Manuals:

Biltz (Jones-King), Determ'n of Mol. Wts. by Boil.-Pt. Methods, p. 141
Briggs, V, Elevation of the Boil.-Pt., p. 17
Ewell, III, Solutions, p. 175
Findlay, VII, Mol. Wt. of Subs. in Solution, p. 147
Firth, V, Determ'n of Mol. Wts., p. 24
Getman, VI, Melting & Boiling Points, p. 77
Gray, Ex. 8, Raising of Boiling Points, p. 49
Pring, Part I. Phys. Chem. Measurements, p. 9
Roth (Cameron), III, Determ'n of Mol. Wts. in Soln's, p. 47
Traube (Hardin), XI, Elevation of Boiling Points, p. 103

Special Texts:

Holleman (Walker), Organic Chemistry, Par. 14, Determ'n of Mol. Wt., p. 18

Journal Articles:

Eykman, Journal de Chimie Physique, 2, p. 47 (1903)
Menzies and Wright, J. A. C. S., 43, p. 2314 (1921) [1]

Apparatus and Chemicals required:

2 iron clamps
2 iron stands
1 glass jacket (length about 40 cm., diameter 4 cm.)
1 Eykman boiling-point vessel, fitting into jacket
1 thermometer (40–100° C.) graduated in tenths of degrees
1 micro-burner+rubber tubing
1 burette+holder
1 bottle containing pure carbon tetrachloride
1 bottle for carbon tetrachloride residues
1 small sample tube with azobenzene [2]

Fig. 12 gives a sketch of the apparatus. The outer glass jacket *A* held vertically by means of an iron ring or a clamp serves the double purpose of boiling vessel and air cooler.

The inner boiling-point vessel *B* (Eykman boiling-point tube) which fits snugly into this mantel, consists of an outer cylinder surrounding and fused at the top to a test tube *C*. A small bore glass tube *D* is fused into the bottom of the test tube and passes upward between the walls of the latter and the cylinder. Vapor of the solvent enters the test tube through this small bore tube and brings the solution to boiling.

The Eykman vessel is suspended from a clamp which also holds the thermometer in a central vertical position.

The thermometer should be graduated in tenths of degrees so that hundredths can be estimated by means of a reading glass.

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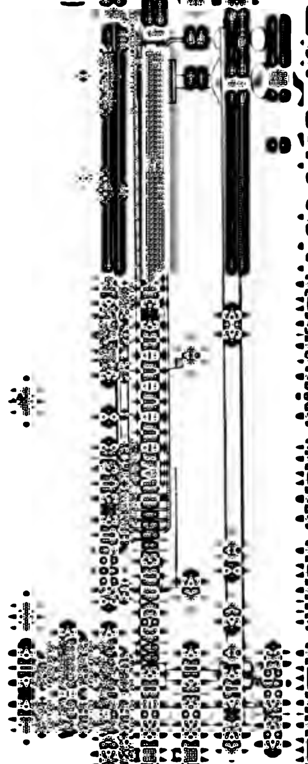
[1] This article discusses the most recent developments of ebullioscopy, and contains a fairly complete bibliography.

[2] CCl_4 is selected for its non-inflammability, and azobenzene for its color, which shows possible residues from a previous experiment at once.



[illegible] the volume of the solution [illegible] necessary first to calibrate [illegible]

[illegible] el until its top comes flush [illegible] of the outer jacket and [illegible] at the inner vessel and the [illegible] are both exactly vertical. [illegible] should always rest on the [illegible] inner tube.

[illegible] burette filled to the zero [illegible] pure carbon tetrachloride, [illegible] number of c.c. into the [illegible] until the liquid rises on the [illegible] scale.

[illegible] e upper meniscus on the [illegible]

[illegible] ctly 1 c.c. at a time, reading [illegible] ach time on the thermometer [illegible] have been added.

[illegible] table which will show the [illegible] the solution in the tube at [illegible] shown on the thermometer.

[illegible] enough of the solvent from [illegible] be to bring the level to the [illegible] he scale and place about 50 [illegible] carbon tetrachloride in the [illegible]

[illegible] at the bottom of the inner [illegible] level of the liquid inside [illegible]

[illegible] outer jacket and adjust the [illegible] apor condenses about 5 cm. [illegible] at *E*, Fig. 12).

[illegible] ery 15 seconds and record [illegible] gives a value constant to [illegible] has been reached.

Raise the inner vessel till its top comes flush with the top of the jacket and add from 0.15 to 0.200 gram of azobenzene, accurately weighed, observing care lest some of it stick to the thermometer or the side of the tube.

Lower the vessel to the same level as before, and determine the boiling point of the solution. Be sure that the solution actually boils; this can readily be seen by observing whether the vapor of the solvent bubbles through the solution. If this does not take place the inner vessel should be lowered a little more.

Raise the tube to its previous height, *observe the volume* using precaution to make sure that conditions are the same as in calibrating. Record volume.

Add successively 3 weighed quantities of azobenzene (0.15–0.200 gram) and note carefully the boiling point and the volume each time, as directed above.

When all the readings have been taken, turn off the gas, remove the thermometer, pour the residue from the vessel into a bottle provided for this purpose and rinse both the thermometer and the tube with a few c.c. of pure carbon tetrachloride, transferring the liquid each time to the residue bottle.

Do not attempt to rinse and clean the apparatus with water.

Observations and Measurements:

Volume readings on thermometer:

.... c.c. =	.... scale reading	.... c.c. =	....
....	....	....	....
....	....	....	....
....	....	....	....
....	....	....	....
....	....	....	....
....	....	....	....
....	....	....	....
....	....	....	....
....	....	....	....

Barometric pressure . . . . mm. Corrected value . . . . mm

Room temperature . . . . °

Boiling point of pure solvent and of 4 solutions, readings every 15 seconds:

	Added . . . gm.	Added . . . gm.	Added . . . gm.	Added . . . gm.
. . . . . .	. . . . . .	. . . . . .	. . . . . .	. . . . . .
. . . . . .	. . . . . .	. . . . . .	. . . . . .	. . . . . .
. . . . . .	. . . . . .	. . . . . .	. . . . . .	. . . . . .
. . . . . .	. . . . . .	. . . . . .	. . . . . .	. . . . . .
. . . . . .	. . . . . .	. . . . . .	. . . . . .	. . . . . .
. . . . . .	. . . . . .	. . . . . .	. . . . . .	. . . . . .
. . . . . .	. . . . . .	. . . . . .	. . . . . .	. . . . . .
. . . . . .	. . . . . .	. . . . . .	. . . . . .	. . . . . .
. . . . . .	. . . . . .	. . . . . .	. . . . . .	. . . . . .
. . . . . .	. . . . . .	. . . . . .	. . . . . .	. . . . . .
. . . . . .	. . . . . .	. . . . . .	. . . . . .	. . . . . .
. . . . . .	. . . . . .	. . . . . .	. . . . . .	. . . . . .
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. . . . . .	. . . . . .	. . . . . .	. . . . . .	. . . . . .
. . . . . .	. . . . . .	. . . . . .	. . . . . .	. . . . . .

Volume readings on thermometer

. . . .

Volume readings in c.c.

. . . .

Calculations:

1. Assuming that the specific gravity of the solutions is practically the same as that of the pure solvent, find the weight of solvent used in each case.

2. Express the four concentrations in terms of grams of solute in 100 grams of solvent [weight of solvent (S) : weight of solute (g) = 100 : x]

3. Record the data obtained : Δ (rise of boiling point), S, g, and concentration in following table:

Solution	Δ	S	g	conc
1	......	......	......	......
2	......	......	......	......
3	......	......	......	......
4	......	......	......	......

4. Apply the boiling-point formula and calculate for each concentration the molecular weight of azobenzene. Obtain boiling-point constant from Table 6, Appendix.

5. Plot the molecular weights obtained as a function of the concentrations and connect the points obtained by a straight line. Where it intersects the Y-axis, the value for the molecular weight at infinite dilution which is the nearest approach to the theoretical value, is obtained.

EXPERIMENT 8

DISTRIBUTION OF A SUBSTANCE BETWEEN TWO NON-MISCIBLE LIQUIDS

Object:

To determine the partition of benzoic acid between water and benzene, and to test the validity of Nernst's distribution law in this case.

References:

Textbooks:

Bigelow, XXIV, Process of Ppt'n & Sol'n, p. 350
Getman, XIII, Heterogeneous Equilibrium, p. 390
Lincoln, XV, Soln's of Liquids in Liquids, I, p. 165
Millard, V, Solutions, p. 117
Morgan, VI, Solutions, p. 201
Senter, VIII, Heterogeneous Equilibrium, p. 177
Walker, XX, Mol. Complexity, p. 216
Washburn, XIV, Distribution Laws, p. 148

Laboratory Manuals:

Ewell, VII, Chemical Statics, p. 262
Findlay, VIII, Dist. of Subs. between 2 Non-misc. Solvents, p. 155
Firth, XI, Determ'n of Part. Coeffs., p. 71
Gray, Ex. 13, Distribution Factor, p. 63

Apparatus and Chemicals required:

3 150-c.c. separatory funnels, labeled 1, 2 and 3 with cocks and stoppers attached by wires
2 125-c.c. wide-mouth Erlenmeyer flasks
1 5-c.c. pipette
1 2-c.c. pipette

1 25-c.c. graduated cylinder
1 iron stand
3 small rings to hold separatory funnels
1 tripod with wire gauze
1 Bunsen burner+rubber tubing
1 bottle containing pure benzoic acid
1 bottle containing benzene
1 bottle containing CO_2-free water
1 large stock bottle containing approximately N/20 baryta solution. This bottle should be fitted with the necessary guard tubes and be connected with a 25-c.c. burette, graduated in twentieths of a c.c.
1 dropping bottle containing phenolphthalein indicator.

Method of Procedure:

Place 25 c.c. of CO_2-free water and 25 c.c. of benzene in each of the three separatory funnels and introduce 1.25, 1.50 and 1.75 grams of benzoic acid respectively into the numbered funnels.

Stopper at once and shake for 15 minutes, holding the funnels by the stem and neck in such a manner that the mixture is not warmed by the hands.

Place the separatory funnels in their supporting rings and allow the mixture to separate into two perfectly clear layers.

Analyze the upper (benzene) layer in each of the funnels as follows, taking care not to warm the liquids:

Place 25 c.c. of CO_2-free water and a drop of phenolphthalein in each of the two 125-c.c. Erlenmeyer flasks. By means of the 2-c.c. pipette withdraw and discard a sample from the upper layer in funnel No. 1, then place exactly 2 c.c. of this liquid in each of the Erlenmeyer flasks.

Heat these solutions to boiling and titrate at once with N/20 baryta solution, being careful not to overrun the end-point. Should the two titrations not check to 0.05 c.c. take further samples and repeat the titrations.

Having analyzed the upper layer in funnel No. 1, analyze those in the other two funnels in like manner.

Carefully draw about 17 c.c. of the lower (aqueous) layer from funnel No. 1, running it into the *dry* graduated cylinder. Rinse the 5-c.c. pipette with this solution, then run exactly 5 c.c. into each of the Erlenmeyer flasks, add 20 c.c. of CO_2-free water and a drop of phenolphthalein to each, heat to boiling and titrate with baryta solution.

After having obtained satisfactory "checks" (to 0.05 c.c.), dry the measuring cylinder and proceed to the analysis of the aqueous layer in the other two funnels.

Before leaving the laboratory, see that the solutions are drained from the separatory funnels. Remove the stopcocks, and leave the funnels supported in their rings so that they will drain and dry before the next laboratory period.

Observations and Measurements:

	No. 1	No. 2	No. 3
Grams of benzoic acid	......	......	......
c.c. of water	......	......	......
c.c. of benzene	......	......	......
Analysis of upper (benzene) layer:			
c.c. of baryta	......	......	......
	......	......	......
	......	......	......
	......	......	......
Mean of constant values	......	......	......
c.c. baryta/c.c. sample T_B	......	......	......
Analysis of lower (aqueous) layer:			
c.c. of baryta	......	......	......
	......	......	......
	......	......	......
	......	......	......
Mean of constant values	......	......	......
c.c. baryta/c.c. sample T_W	......	......	......

Calculations:

1. Express the numerical values of the ratio $\frac{T_W}{T_B}$ for the three solutions and see whether a constant value is obtained or not. If not, what conclusion can be drawn?

2. Calculate the ratios: $\frac{\sqrt{T_W}}{T_B}$ and $\frac{T_W}{\sqrt{T_B}}$. If either of these is constant what inference can be drawn?

EXPERIMENT 9

DISTILLATION WITH STEAM

Object:

To illustrate the principle of steam distillation, and to show that the molecular weight of the less volatile constituent can be determined from the partial pressures of the distilling vapors and the weights of the liquids obtained in the distillate.

References:

Textbooks:

Bigelow, XXIV, Process of Solution & Ppt'n, p. 333
Getman, VII, Solutions, p. 250.
Lincoln, XVI, Soln's of Liquids in Liquids, II, p. 181
Millard, V, Solutions, p. 133
Senter, IV, Solutions, p. 90
Walker, VIII, Vaporiz'n & Condensation, p. 80
Washburn, XVI. Solutions, IV, p. 181

Special Texts:

Holleman (Walker), Organic Chemistry. Par. 23, Steam Distillation, p. 26
Robinson, The Elements of Fractional Distillation. Table 3, Appendix, pp. 180–195

Apparatus and Chemicals required:

2 iron stands
2 clamps
1 tripod
1 wire gauze
1 metal steam generator
1 one-liter round bottom flask

1 connecting side-arm still head
1 thermometer (50–100°) graduated in tenths of a degree
1 30-inch condenser+rubber tubing
2 Bunsen burners+rubber tubing
3 100-c.c. graduates
1 adapter
1 separatory funnel
1 250-c.c. Erlenmeyer flask
1 500-c.c. bottle containing either chlorobenzene (B. P. 132°), bromobenzene (B. P. 155°) or nitrobenzene (B. P. 210°)

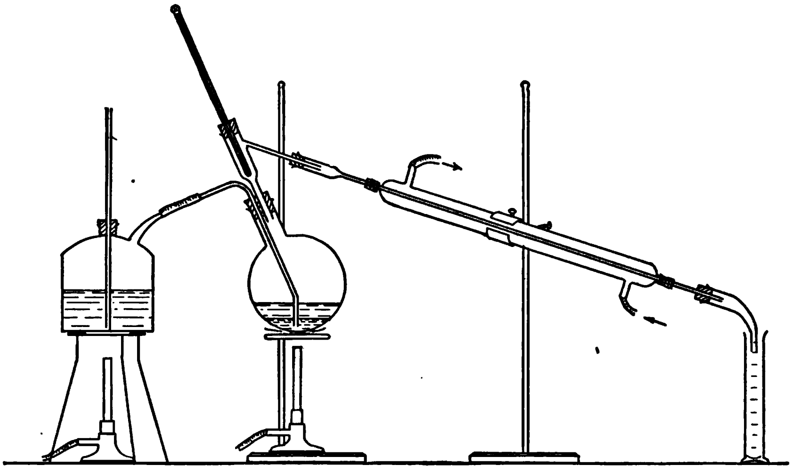


Fig. 13.

Fig. 13 represents the apparatus used for steam distillation. A round-bottom flask inclined at an angle is supported on an iron ring and closed by a two-hole cork. Through one hole passes a glass tube bent downward so as to reach within 0.5 cm. of the bottom of the flask, while the other hole bears the still head which connects the flask to the condenser. A thermometer reading to tenths of a degree is inserted in the still head. Steam is generated in the metal can on the left. This can is provided with a safety tube which reaches to the bottom and extends about 40 cm. above the top.

Method of Procedure:

Half fill the steam generator with water, close the neck by the one-hole cork carrying the safety tube and connect the steam outlet pipe with the round-bottom flask.

Place in the flask 300 c.c. of the organic liquid to be used together with 25 c.c. of water, make all connections tight and see that cold tap water is running through the condenser.

Heat both the can and the flask, the latter by means of a *low* flame which may be removed as soon as the flask is thoroughly heated and the steam no longer condenses in it.

In order to prevent the partial condensation of vapor in the neck of the flask and the still head, wrap a piece of cloth around these parts.

Record the barometric pressure and the room temperature at the beginning and the end of the experiment.

Collect the first 10–20 c.c. of the distillate separately in an Erlenmeyer flask and as soon as the distillate comes over regularly collect three 100-c.c. samples in the graduated cylinders. Watch the temperature carefully during the time of sample taking and record the same every minute, for the distillation should be carried out at constant temperature. This will be the case so long as the steam bubbles through the organic liquid at a uniform rate.

Leave the distillates in the graduates until the next laboratory period when the emulsions will have changed into clear layers. Both the upper (aqueous) and the lower layer in each graduate are then carefully measured in c.c.

Disconnect the round-bottom flask, cool and empty its contents into the separatory funnel. Replace the empty flask and pass steam through it, recording one-minute temperature readings until five consecutive constant observations are obtained. Knowing the correct barometric pressure a correction for the boiling points of pure water and the mixture can be applied.

Turn off both gas and water before leaving the laboratory.

Observations and Measurements:

Barometric pressure at start: ....mm. corrected value..
room temperature: ....°
Same at end of experiment: ....mm. corrected value..
room temperature: ....°

Observed boiling point of mixture (readings every minute)

....
....
....
....
....
....
....

Value of boiling-point of mixture as derived from these observations ......°

Observed boiling-point of water:

....
....
....

Corrected values: b.p. mixture ....°, of water ....°.

	Cyl. 1	Cyl. 2	Cyl. 3	Total
Number of c.c. of water collected	......	......	......	......
Number of c.c. of .... collected	......	.. ....	......	......

Room temperature ....°

Calculations:

1. From the densities of the liquids, for which see Table 4, calculate the weight of each.

2. Look up in Table 2 the vapor pressure of water at the (corrected) boiling point of the mixture, and ascertain the partial pressure of the organic liquid.

3. From these data and the molecular weight of water find the (approximate) molecular weight of the organic liquid used in this experiment.

EXPERIMENT 10

MINIMUM BOILING LIQUIDS

Object:

To show that mixtures of certain pairs of liquids have boiling points which are lower than the boiling points of either of the pure compounds.

References:

Textbooks:

Bigelow, XXIII, Vapor Press. of Soln's, p. 309
Getman, VII, Solutions, p. 202
Jones, V, Solutions, p. 183
Lincoln, XVI, Soln's of Liq. in Liq., II, p. 179
Millard, V, Solutions, p. 132
Morgan, VI, Solutions, p. 137
Senter, IV, Solutions, p. 87
Walker, VIII, Vaporiz'n & Condensation, p. 78

Laboratory Manuals:

Briggs, VII, Distil'n of Liquid Mixtures, p. 26
Ewell, III, Solutions, p. 157

Special Texts:

Robinson, l. c., Table 3, Appendix, pp. 180–188

Apparatus and Chemicals required:

1 three-necked boiling-point vessel
1 iron stand
1 clamp
1 iron ring

1 tripod
1 wire gauze
1 perforated asbestos plate
1 20-inch condenser+rubber tubing
1 Bunsen burner+rubber tubing
1 thermometer (0–100°)
1 graduated cylinder (50 c.c.)
1 liter bottle containing benzene
1 liter bottle containing methyl alcohol

Fig. 14 gives a sketch of the assembled apparatus. The three-necked boiling-point vessel is supported on the wire gauze by means of the perforated asbestos plate (laid on the iron ring) and connected air-tight to the reflux water-cooler on the right. The central opening of the vessel is closed by a tight-fitting perforated cork carrying a thermometer.

Method of Procedure:

Since both benzene and methyl alcohol are highly inflammable it is necessary to insure air-tight connections especially at the one-hole cork connecting the boiling-point vessel to the condenser.

Regulate the flow of tap water through the condenser. Pour 50 c.c. of benzene measured in the 50-c.c. graduate, through the side neck on the left.

Close the neck and determine the boiling point of the liquid. In this and *all* following determinations the bulb of the thermometer should dip *in the liquid* and the thermometer be of such length that the cork does not interfere with readings above 55°.

Record the barometric pressure and room temperature.

Having obtained the boiling point, turn off the gas, wait a few minutes until the liquid has sufficiently cooled, and introduce 10 c.c. of methyl alcohol through the side neck.

Close the neck and determine the boiling point of this mixture.

Repeat the operation twice by adding successively 20 and 30 c.c. of methyl alcohol.

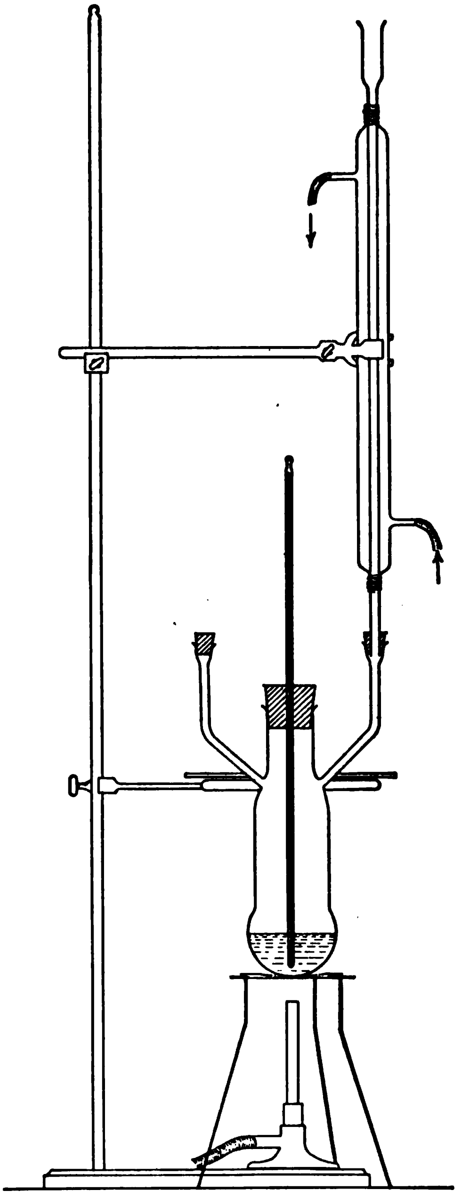


FIG. 14.

Disconnect flask from condenser and transfer the solution to the waste bottle. Rinse the flask with about 10 c.c. of methyl alcohol and after assembling the apparatus as before, introduce 50 c.c. of methyl alcohol and determine its boiling point.

Repeat the boiling-point determination three times by adding successively 10, 20 and 25 c.c. of benzene.

Turn off both the gas and water supply and pour the contents of the flask into the waste bottle. Do not rinse the boiling-point vessel with water.

Tabulation of Results:

Barometric pressure: . . . .mm. Corrected value: . . . .mm.
Room temperature: . . . .°
Liquid A (benzene)
Liquid B (methyl alcohol)

A	B	Boiling point (uncorrected)
50 c.c.		. . . . . .
50 c.c.	Added 10 c.c.	. . . . . .
50 c.c.	20 c.c. (total 30)	. . . . . .
50 c.c.	30 c.c. (60)	. . . . . .
B	**A**	
50 c.c.		. . . . . .
50 c.c.	Added 10 c.c.	. . . . . .
50 c.c.	20 c.c. (total 30)	. . . . . .
50 c.c.	25 c.c. (55)	. . . . . .

Calculations:

1. Compute the volumes of A and B for each mixture, on the basis of a total volume of 100 c.c.

2. Prepare a temperature-concentration diagram by plotting the temperature as ordinate (10° = 5 cm.) against the volume concentration (10 per cent = 1 cm.) as abscissa.

3. Construct a smooth curve through these 8 points, designated as P_1, P_2, etc., and locate the minimum on the curve.

EXPERIMENT 11

BOILING-POINT AND VAPOR COMPOSITION CURVE OF LIQUID MIXTURES

Object:

To determine the boiling-point and vapor composition curve for the binary liquid mixture (maximum boiling point): hydrochloric acid-water.

References:

Textbooks:

Bigelow, XXIII, Vapor Press. of Solutions, p. 307
Getman, VII, Solutions, p. 201
Lincoln, XVI, Soln's of Liquids in Liquids, II, p. 178
Millard, V, Solutions, p. 120
Senter, IV, Solutions, p. 89
Walker, VIII, Vaporiz'n & Condensation, p. 79

Laboratory Manuals:

Briggs, VII, Distill'n of Liquid Mixtures, p. 23
Ewell, III, Solutions, p. 157

Special Texts:

Holleman (Walker), Textbook of Organic Chemistry, Par. 22, Fractional Distillation, p. 24
Robinson, Fractional Distillation, Table 3, Appendix. p. 184 (with litt. ref.)

Apparatus and Chemicals required:

1 1000-c.c. distilling flask
1 30-inch condenser
1 thermometer (100–150°) graduated in tenths of degrees

3 iron stands
2 clamps
1 burette holder
1 burette
2 test-tube racks with 24 stoppered and labeled test tubes.
1 5-c.c. pipette
1 1-c.c. pipette
1 tripod
1 wire gauze
2 Bunsen burners+rubber tubing
1 100-c.c. measuring cylinder
1 150-c.c. Erlenmeyer flask
1 wash bottle with distilled water
1 stock bottle containing a 10 per cent solution of HCl
1 stock bottle containing fifth normal NaOH
Phenolphthalein indicator
Pumice stone or glass beads

The apparatus is set up as for an ordinary distillation. The flask should be a long-necked one so that the thermometer held by a tight-fitting cork is completely immersed in the vapor over the scale readings from 100 to 110°. The bulb of the thermometer should not dip in the liquid but extend about 1 inch below the side neck of the flask

Method of Procedure:

See that the test tubes in which the samples are to be collected are dry and clean. They are provided with corks and labeled D_1, D_2, D_3, etc., and R_1, R_2, R_3, etc., in order to distinguish the samples of distillate from samples of residue.

Place 500 c.c. of the 10 per cent solution of hydrochloric acid in the distilling flask with a few bits of pumice stone or glass beads to prevent excessive bumping. Violent bumping can further be obviated by gently rocking the stand and flask to and fro, thereby swirling the liquid about in the flask. The use of an asbestos mat or a wire gauze increases bumping and for this reason the distillation should be carried out with a bare flame.

Adjust the flame in such a manner that distillation proceeds rather vigorously, yet at a uniform rate. As soon as the thermometer has warmed up to equilibrium, i.e., after from 10 to 15 c.c. of distillate have been collected in the graduated cylinder, place the test tube labeled D_1 at the outlet of the condenser and collect about 4 c.c. of distillate. While this portion is being distilled read the thermometer to 0.1° and record the same.

When enough distillate has been collected, remove the flame, pipette off a 5-c.c. sample of the liquid in the flask (this portion need not be measured accurately) and place it in the test tube marked R_1. Stopper the test tubes immediately.

Replace the thermometer and continue the distillation (using the same flame) at the same rate as before until the thermometer shows a rise of from 0.8 to 1.0°. Reject the portion which has come over during this interval, then collect a 4-c.c. sample of distillate in tube D_2, taking a temperature reading and recording the same as before. Remove the flame and place a 5-c.c. sample from the flask in Tube R_2.

Continue the operation as outlined above, taking (approximately) 4-c.c. samples of distillate and 5-c.c. samples of residue and recording the temperature, for each rise of approximately one degree, until about 10 c.c. of liquid remain in the flask, when a final sample of distillate and residue are taken and the final temperature read.

While one student is carrying out the distillation, his partner determines the HCl contents of the samples by titrating the same with standard (N/5) alkali. The series of *R*-samples should be cooled to the same temperature as the *D*-samples before analysis.

The analysis is carried out by withdrawing exactly 1 c.c. of sample by means of the small pipette, running it into a 150-c.c. Erlenmeyer flask, adding about 25-c.c. of boiling distilled water from a wash bottle heated over a wire gauze, and titrating with standard alkali (using two drops of phenolphthalein as indicator) until the first permanent pink color develops. Before taking a sample from a test tube rinse the pipette with the solution to be sampled and remove the last drop of rinse solution from the tip by touching it with a piece of filter paper. Each titration is run in duplicate and the result entered in the record of observations.

When the distillation is complete, rinse the distilling flask, introduce about 250 c.c. of distilled water, assemble the apparatus as before and check the reading of the thermometer at the boiling point of pure water. Record the barometric pressure, then obtain the true boiling point of water at this pressure from Table 2 in the Appendix, and if the thermometer does not read correctly, determine the correction to be applied. Correct all observed temperature readings.

Observations and Measurements:

	Temperatures		c.c. of alkali used			Normality	Percentage HCl
	Observed	Corrected	Sample 1	Sample 2	Average		
D_1	....	....	....	....	....	....	....
R_1			....	....	....	....	....
D_2	....	....	....	....	....	....	....
R_2			....	....	....	....	....
D_3	....	....	....	....	....	....	....
R_3			....	....	....	....	....
D_4	....	....	....	....	....	....	....
R_4			....	....	....	....	....
D_5	....	....	....	....	....	....	....
R_5			....	....	....	....	....
D_6	....	....	....	....	....	....	....
R_6			....	....	....	....	....
D_7	....	....	....	....	....	....	....
R_7			....	....	....	....	....

Barometric pressure, ...mm. Room temperature....°
Same corrected ....mm.
Boiling point of water as found ....°
Same as given by table, ...°, Correction: ....°

Calculations:

1. From the result of the titrations find the normality of each sample of distillate and residue, and record the same in the above table.

2. From Table 7 in the Appendix, find the percentage of HCl corresponding to each normality, and place these values in the last column.

3. Plot, on a sufficiently large scale, the percentages so found as abscissæ against temperatures as ordinates. Connect the points by curves and locate the position of the maximum boiling mixture.

EXPERIMENT 12

SOLUBILITY CURVE FOR A TERNARY SYSTEM OF LIQUIDS

Object:

To determine the solubility curve for a ternary system of two non-miscible liquids (water and benzene) and a third liquid (acetic acid)[1] consolute with either of the two.

References:

Textbook:

Lincoln, XV, Solutions of Liquids in Liquids, p. 161

Laboratory Manual:

Briggs, XV, Equil. & Phase Rule, p. 65

Special Text:

Findlay's Phase Rule, 3d Ed., XIV, Soln's of Liquids in Liquids, p. 243

Journal Articles:

Lincoln, Jour. Phys. Chem. 4, p. 161; 8, p. 248.

Apparatus and Chemicals required:

2 iron stands
2 burette holders
4 burettes
1 5-c.c. pipette
1 2-c.c. pipette
1 125-c.c. Erlenmeyer flask + cork stopper
1 300-c.c. Erlenmeyer flask + cork stopper
1 bottle containing distilled water
1 bottle containing benzene
1 bottle containing glacial acetic acid

---

[1] Benzene may be replaced by chloroform, if desired, with equally good results.

Method of Procedure:

(*Student A.*) Using the 5-c.c. pipette, introduce 5 c.c. of benzene into the 125-c.c. Erlenmeyer flask, then add 1 c.c. of water from one burette, and from another burette, little by little, glacial acetic acid until after vigorous shaking a clear (non-cloudy) solution is obtained which is saturated with respect to the three components.

Record the volumes added.

Add 2 c.c. of water to the liquid, shake vigorously and repeat the addition of acetic acid until the mixture again becomes homogeneous.

Add successively 3, then 4, 5 and finally 6 c.c. of water and in each case add enough acetic acid to produce a clear liquid. Record all results.

(*Student B.*) By means of the 2-c.c. pipette introduce 2 c.c. of benzene into the 300-c.c. Erlenmeyer flask. Add 25 c.c. of water from one burette and then from a second, enough glacial acetic acid to produce a clear homogeneous liquid after shaking.

The operation is repeated by adding successively 5 more portions of 25 c.c. of water; in each case the exact volume of acetic acid necessary to produce a clear, noncloudy solution is recorded.

Tabulation of Results:

	Benzene	Water				Acetic Acid				Total vol. in c.c.
A_1	5		1				....			....
A_2	5	Added	2	Total	....	Added	....	Total	....	....
A_3	5		3		....		....		....	....
A_4	5		4		....		....		....	....
A_5	5		5		....		....		....	....
A_6	5		6		....		....		....	....
B_1	2		25				....			....
B_2	2	Added	25	Total	....	Added	....	Total	....	....
B_3	2		25		....		....		....	....
B_4	2		25		....		....		....	....
B_5	2		25		....		....		....	....
B_6	2		25		....		....		....	....

Calculations:

1. Assuming that there is no volume change on mixing, compute the volumes of the three components in equilibrium on the basis of having in each case a total volume of 100 c.c.

2. Plot these percentages upon a triangular diagram, indicating the points by A_1, A_2 ....; B_1, B_2 ...., etc. Connect these 12 points by a smooth curve which is to be prolonged by a dotted line ending in the vertices of the triangle, representing 100 per cent water and 100 per cent benzene respectively (it is assumed that benzene and water are completely immiscible).

3. Recalculate the results, expressing the volumes of benzene and water used, in terms of 100 c.c. of acetic acid and plot these data on ordinary cross-section paper.

EXPERIMENT 13

TRANSITION POINTS IN THE SOLID STATE

Object:

To determine the transition point of anhydrous sodium sulphate by the thermometric method.

References:

Textbooks:

Bigelow, XXI, Vapor Pressure, p. 292
Jones, IX, Chem. Dyn. & Equil., p. 589
Lincoln, XVII, Phase Rule, p. 190
Senter, VIII, Hetereogeneous Equil., p. 183
Walker, X, Phase Rule, p. 101
Washburn, V, Crystalline State of Aggregation, p. 66

Laboratory Manuals:

Briggs, XV, Equil. & Phase Rule, p. 61
Ewell, VII, Chem. Statics, p. 276
Findlay, XV, Determ'n of Trans. Pts., p. 308
Firth, VI, Determ'n of Trans. Pts., p. 35
Getman, XV, Chem. Kinetics, p. 248
Gray, Ex. 12, Determ'n of Trans. Pts., p. 60
Roth (Cameron), VII, Chem. Statics & Dynamics, p. 104

Special Work:

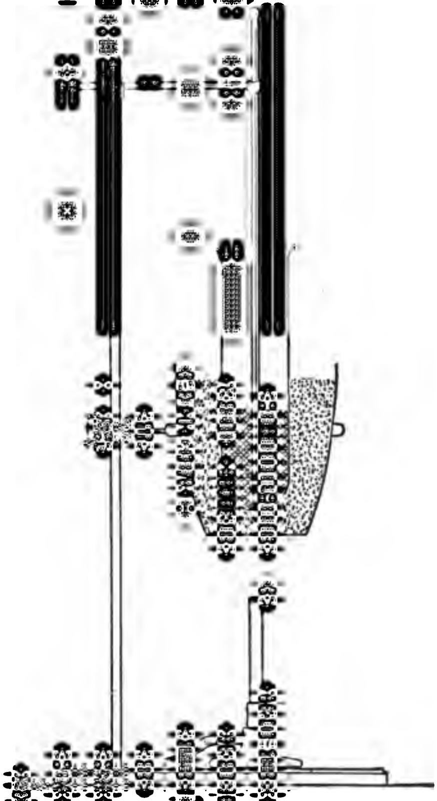
Findlay's Phase Rule, 3d Ed., p. 341.

Apparatus and Chemicals required:

1 iron stand
1 clamp
1 iron ring
1 large copper or iron crucible (dimensions 8–10 cm.)
1 test tube (length 15 cm., diameter 3.5 cm.)
1 Bunsen or Méker burner+rubber tubing

[illegible]

[illegible] as sodium sulphate [illegible]



[illegible]

[illegible] rstood from Fig. 15. The [illegible] iron ring and contains dry [illegible] the test tube. The ther- [illegible] inside the tube by a clamp, [illegible] etely immersed in the finely [illegible]

[illegible]

[illegible] as indicated above the [illegible] unsen or Méker burner and the temperature of the thermometer registered at intervals of half a minute starting from 150° upward.

As soon as the temperature reaches 300 , the gas is turned off and on cooling the temperature registered from 300° to 150°.

Thermometer Readings at Half-Minute Intervals:

On heating				On cooling			
....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....

Observed transition range:

....	....

Plotting the Results:

1. Plot the temperature as ordinate (10° = 1 cm.) against the time as abscissa and indicate by means of an arrow the temperatures of maximum heat absorption and evolution on the heating and cooling curve respectively.

In order to locate (inflexion) points more accurately choose the unit of time on the horizontal axis of such length that the slope of the curve as a whole is under an angle of 45° to horizontal.

Due to the well-known phenomenon of hysteresis these temperatures do not coincide and the true inversion or transition temperature lies somewhere in between.

EXPERIMENT 14

PHASE DIAGRAM FOR A TWO-COMPONENT SYSTEM OF METALS

Object:

To determine by means of cooling curves the conditions of equilibrium in a binary alloy.

References:

Textbooks:

Bigelow, XXIV, Process of Sol'n & P'p't'n, p. 349
Getman, XIII, Heterogeneous Equil., p. 414
Lincoln, XIX, Soln's of Solids in Liquids, II, p. 211
Millard, IX, Heterogeneous Equil., p. 278
Senter, VIII, Hetereogenous Equil., p. 190
Walker, XI, Alloys, p. 112

Laboratory Manual:

Ewell, VII, Chem. Statics, p. 271

Apparatus and Chemicals required:

- 2 iron stands
- 2 small iron rings
- 2 Bunsen burners+rubber tubing
- 2 clamps
- 2 thermometers (nitrogen-filled) graduated to 400°.
- 6 Battersea crucibles D (diameter 6 cm., length 10 cm.), numbered from 1 to 6 containing respectively:
    - 200 grams of cadmium
    - 160 grams of cadmium+40 grams of bismuth
    - 120 grams of cadmium+80 grams of bismuth
    - 80 grams of cadmium+120 grams of bismuth
    - 40 grams of cadmium+160 grams of bismuth
    - 200 grams of bismuth
- 1 small bottle with powdered charcoal
- 1 small bottle with nitric acid

One unit of the apparatus is shown in Fig. 16. The metal or alloy under observation is placed in a Battersea crucible and covered with a thin layer of charcoal to prevent oxidation. The crucible is supported in an iron ring and heated over a Bunsen burner. The thermometer, covered at the top with a piece of heavy rubber tubing, moves freely through a perforated cork held by a clamp and sinks by its own weight in the metal (or alloy) as soon as the latter fuses.

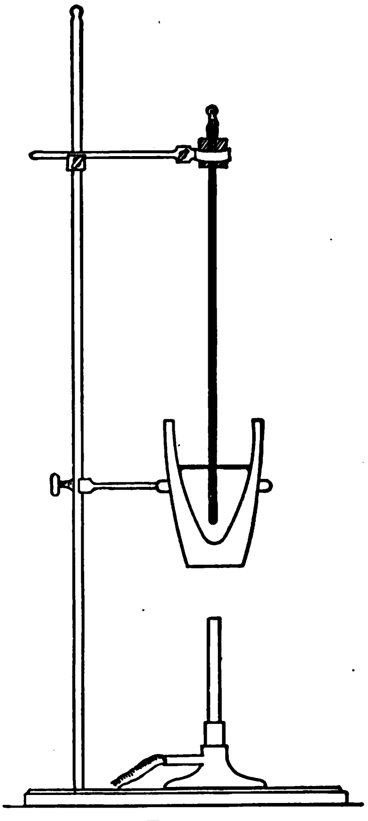


FIG. 16.

Method of Procedure:

Student A confines his observations to samples 1, 2 and 3; student B to samples 6, 5 and 4.

Barely melt the metal in crucible 1(6) and see that the bulb of the thermometer is immersed in the center of the molten metal. Be careful that the mercury does not rise above 400°. If it does tend to rise above this limit, immediately withdraw the thermometer and do not return it to the melt until the temperature has fallen to below 400°.

Remove the flame, allow the system to cool without interference of any kind (avoid air drafts!) and record temperature readings at 15-second intervals over the range, 340°–120°.

Again melt the metal taking temperature readings from 130°–330°. As soon as the thermometer reaches the latter temperature remove the thermometer.

When the mercury has fallen to below 100° remove any adhering metal by dissolving it in nitric acid in order to prevent a change in the composition of the next melt.

In a similar manner obtain cooling (and heating) curve data for the alloys in crucibles 2 and 3 (5 and 4). In these cases take readings between the limits 300°–120° as the alloys fuse at a much lower temperature than the pure metals.

Observations:

Sample 1 or 6			2 or 5			3 or 4		
				On Cooling				
....	....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....	....
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....	....	....	....	....	....	....	....	....
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....	....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....	....

On Heating

....	....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....	....
....	....	....	....	...	....	....	....	....
....	....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....	....
....	....	....	....	....	...	....	....	....

Plotting the Results:

1. Plot all three cooling curves on the same sheet of coordinate paper (temperature as ordinate vs. time as abscissa); by shifting the time axis successively 2 cm. to the right. Locate for each sample the temperature of initial and final (eutectic) solidification and unite these observations in a table giving for each of the six samples the exact composition and the temperatures of initial and eutectic solidification.

2. Use the data thus obtained for the construction of a complete temperature-composition diagram (plot temperatures vertically and compositions horizontally).

3. On a separate sheet plot the heating curves and see how the breaks in these curves check with those obtained from the cooling curves.

4. In case a metallographic microscope is available, observe the structures of the pure metals, and of the four alloys, on polished and etched samples having the above indicated compositions.

EXPERIMENT 15

MONOMOLECULAR REACTION: INVERSION OF CANE SUGAR

Object:

To determine the reaction velocity for the monomolecular reaction: the inversion of cane sugar.

References:

Textbooks:

Bigelow, XXV, Vel. of Chem. Reactions p. 353
Getman, XIV, Chem. Kinetics, p. 428
Jones, IX, Chem. Dyn. & Equil., p. 531
Lincoln, XXXVI, Rate of Chem. Reactions, p. 487
Millard, X, Kinetics of Reactions, p. 299
Morgan, VIII, Chemical Change, p. 282
Senter, IX, Vel. of Reaction, p. 229
Walker, XXV, Rate of Chem. Trans., p. 286
Washburn, XXI, Chem. Kinetics, p. 270

Laboratory Manuals:

Ewell, VI, Chem. Kinetics, p. 232
Findlay, XII, Vel. of Chem. Reactions, p. 262
Firth, XVI, Vel. of Chem. Reactions, p. 140
Getman, XV, Chem. Kinetics, p. 243
Gray, Ex. 28, Vel. of Inv. of Cane Sugar, p. 122
Roth (Cameron), VII, Chem. Statics & Dynamics, p. 106

Apparatus and Chemicals required:

1 polarimeter
1 sodium light
1 jacketed polarimetric tube 20 cm. long
1 coiled copper tube, 1 meter long, internal diameter 5 mm.
1 iron stand
1 clamp

2 Bunsen burners+rubber tubing
1 bottle with three-holed stopper carrying 2 glass tubes and 1 thermometer (0–100°)
1 25-c.c. pipette
2 100-c.c. beakers
1 stock bottle containing normal hydrochloric acid
1 stock bottle containing a 20 per cent sugar solution preserved by a tiny crystal of camphor.

The polarimeter (drawn schematically), jacketed tube, and sodium light are shown in Fig. 17. Tap water is passed at a regulated rate through a coiled copper tube heated by a carefully regulated flame, next through a one-liter bottle containing the thermometer (not shown in the figure) then through the jacketed polarimeter tube, and thence to waste.

A thoroughly satisfactory source of sodium light[1] consists of a small alundum crucible (R.A. 360) about one-third full of NaCl, heated over a Méker burner.

Method of Procedure:

Bring the water circulating through the jacketed tube to 30° and maintain it there by constant watching.

Dry the polarimeter tube.

Place the sodium flame about 20 cms. from the polariscope, light the gas, remove the tube from its rack. and see that the polariscope is in adjustment.

Place 25 c.c. of the 20 per cent sugar solution in one beaker, and an equal volume (use pipette both times) of normal hydrochloric acid in the other. Bring the temperature of each to 30°, mix the solutions and fill the polarimeter tube, taking care to eliminate any air bubbles which might tend to collect therein.

Place the tube in position and at once take a reading, noting the time at which the reading was taken.

For the first twenty minutes take one-minute readings, for the next twenty, two-minute readings and for the next sixty, five-minute readings on the polarimeter, recording also time (and temperature).

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[1] Fales & Morrell, Jour. Amer. Chem. Soc., **43**, p. 1629 (1921).

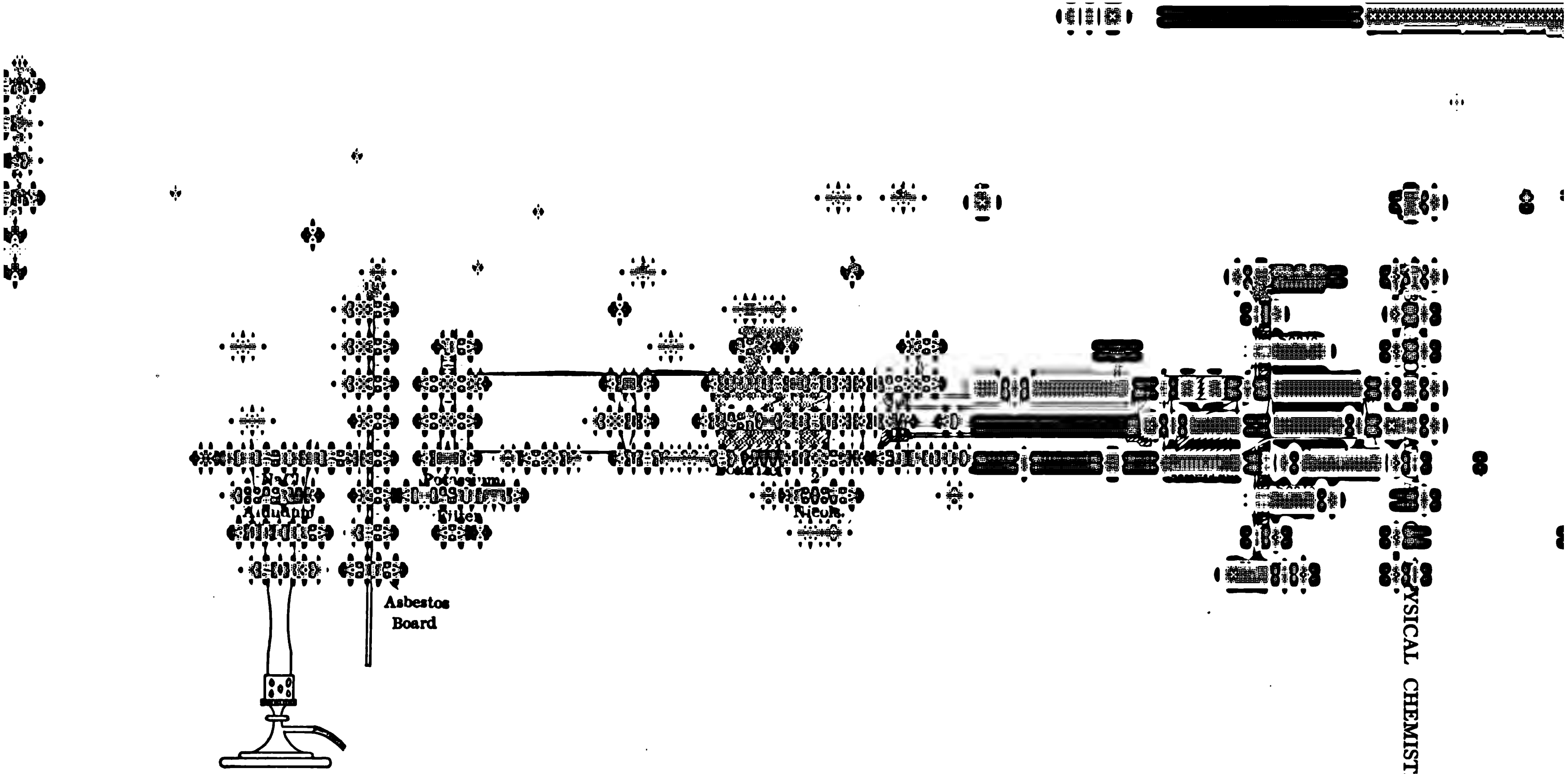

FIG. 17.

At the end of one hundred minutes, raise the temperature of the circulating water to 65° and take one-minute readings until five successive constant values have been obtained; then bring the temperature back to 30° and continue to take one minute readings until five consecutive constant values have again been obtained. At the temperature of 65° the reaction runs to completion quite rapidly and the values at 30° after complete inversion represent the value A_{∞}.

Observations and Measurements:

Readings at 30°.

Angle	Time	Temp.	Angle	Time	Temp.
......	......	......	......	......	......
......	......	......	......	......	......
......	......	......	......	......	......
......	......	......	......	......	......
......	......	......	......	......	......
......	......	......	......	......	......
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......	......	......	......	......	......
......	......	......	......	......	......
......	......	......	......	......	......
......	......	......	......	......	......

Readings at 65°			Final readings at 30°		
......	......	......	......	......	......
......	......	......	......	......	......
......	......	......	......	......	......
......	......	......	......	......	......
......	......	......	......	......	......
......	......	......	......	......	......
......	......	......	......	......	......
......	......	......	......	......	......
......	......	......	......	......	......
......	......	......	......	......	......

Calculations:

1. Plot the observed angles (1° = 1 large division) against the times (5 min. = 1 large division) as abscissae and draw a smooth curve through the points.

2. Select four representative points on the curve and calculate the velocity constant for these points.

EXPERIMENT 16

BIMOLECULAR REACTION: SODIUM THIOSULPHATE REACTING WITH ETHYL BROMACETATE

Object:

To determine the reaction velocity for the bimolecular reaction:

$$CH_2BrCOOC_2H_5+Na_2S_2O_3=CH_2(NaS_2O_3)COOC_2H_5+NaBr$$

References:

Textbooks:

Bigelow, XXV, Vel. of Chem. Reactions, p. 356
Getman, XIV, Chem. Kinetics, p. 429
Jones, IX, Chem. Dyn. & Equil., p. 546
Lincoln, XXXVI, Rate of Chem. React., p. 484
Millard, X, Kinetics of Reactions, p. 303
Morgan, VIII, Chem. Change, p. 289
Senter, IX, Vel. of Reaction, p. 231
Walker, XXV, Rate of Chem. Trans., p. 289
Washburn, XXI, Chem. Kinetics, p. 271

Laboratory Manuals:

Ewell, VI, Chem. Kinetics, p. 243
Findlay, XII, Vel. of Chem. React., p. 270
Firth, XVI, Vel. of Chem. React., p. 141

Journal Article:

Slator, Trans. Chem. Soc., *87*, p. 484 (1905)

Apparatus and Chemicals required:

1 thermostat, 25°±0.1°
1 iron stand
2 burettes
1 burette holder
1 50-c.c. pipette
1 25-c.c. pipette
1 125-c.c Erlenmeyer flask, wide mouth
2 250-c.c. graduated flasks
1 1000-c.c. flask
1 100-c.c. beaker
1 bottle of ethyl bromacetate
1 stock bottle of hundredth normal iodine solution
1 stock bottle of sixtieth normal sodium thiosulphate solution
1 bottle of starch indicator solution

Method of Procedure:

Secure from the instructor a small glass-stoppered vial, containing not over 0.5 gram of ethyl bromacetate, open it under water, in a small beaker, and transfer to a clean 250-c.c. graduated flask.[1] Rinse the beaker with water, and add the washings to the graduated flask. Make up to 250 c.c., and place the flask in the thermostat.

Fill the other 250-c.c. flask to the mark with sodium thiosulphate solution, and place it in the thermostat.

When the temperature of both solutions has reached 25°, the two liquids are mixed in the 1000-c.c. flask (the time of mixing being taken), the flask is shaken, and placed in the thermostat.

At the end of five minutes (counted from the time of mixing) remove 50 c.c. from the reaction flask, and titrate *rapidly* with the hundredth normal iodine solution, using starch as indicator.

At the end of 10, 15, 25, 40, 60, and 90 minutes (from the start of the reaction), withdraw 50 c.c. from the flask, and titrate rapidly as before. Note the time at which iodine solution is run into the titration flask in each instance.

---

[1] The ester has strong lacrymatory properties.

Between the last two titrations, determine the initial concentration of sodium thiosulphate in the flask by withdrawing 25 c.c. from the stock bottle, and titrating with iodine. Why withdraw 25 c.c. here, instead of the 50 taken for the other titrations? Express this concentration in c.c. of the iodine required.

After at least five or six hours, or better still, at the start of the next laboratory period, titrate 50 c.c. of the liquid remaining in the reaction flask. The reaction being presumably complete at this time, the volume of iodine solution required for this titration represents the excess of thiosulphate originally present in the mixture. The value of this titration, therefore, subtracted from the value of the titration of the 25 c.c. of thiosulphate withdrawn from the stock bottle, gives the initial concentration of the ethyl bromacetate solution present in 50 c.c. of the mixture, expressed in c.c. of iodine solution.

Observations and Measurements:

	Time	c.c. iodine
The solutions were mixed at	....	
1st Sample titrated at	....	....
2d “ “ “	....	....
3d “ “ “	....	....
4th “ “ “	....	....
5th “ “ “	....	....
6th “ “ “	....	....
7th “ “ “	....	....
Titration of 25 c.c. stock thiosulphate		....
“ “ mixture at end of reaction		....

Calculations:

1. Using the integrated equation for the velocity constant of bimolecular reactions, calculate the value of “*k*” for each mixture, expressing the concentration in c.c. of iodine solution used.

2. Also calculate the value of the constant, expressing the concentrations of the reacting mixture in moles per liter.

3. Find the time necessary to convert half of the ester.

EXPERIMENT 17

HEAT OF NEUTRALIZATION

Object:

To determine the heat of neutralization of an acid by a base.

References:

Textbooks:

Bigelow, XXVII, Thermochem. Data, p. 406
Getman, XI, Thermochemistry, p. 355
Jones, VI, Thermochemistry, p. 333
Lincoln, XXXIV, Thermochemistry, p. 431
Millard, VII, Thermochemistry, p. 206
Morgan, VII, Thermochemistry, p. 229
Senter, XI, Equil. in Electrolytes, p. 283
Walker, XXIX, Applications of the Diss'n Theory, p. 336
Washburn, XIX, Thermochemistry, p. 243

Laboratory Manuals:

Briggs, XVII, Thermochemistry, p. 84
Ewell, IV, Thermochemistry, p. 193
Findlay, XIII, Thermochemistry, p. 278
Firth, XII, Thermochem. Meas., p. 74
Getman, VII, Calorimetry, p. 107
Gray, Ex. 18, Heat of Neut., p. 80
Pring, Part I, Calorimetry, p. 23
Roth (Cameron), IV, Thermochemistry, p. 59
Traube (Hardin), XV, Thermochem. Constants, p. 133

Apparatus and Chemicals required:

1 mixture calorimeter
1 thermometer (0–50°) graduated to tenths of a degree
1 Beckmann thermometer
2 250-c.c. graduated flasks calibrated to deliver
1 reading glass
1 stock bottle with N/4 sodium hydroxide
1 stock bottle with N/4 hydrochloric acid

The calorimetric vessel is represented in Fig. 18, and consists of a highly polished cylindrical metallic vessel with cardboard or ebonite cover through which pass the Beckmann thermometer, the stirrer, and a glass tube for introducing the solution from the upper vessel. The upper vessel carries a quick-opening valve at the bottom. The calorimetric vessel is surrounded by three other cylindrical vessels and the annular space between the two outer cylinders is filled with water. The upper vessel can be swung into position for discharging its contents into the calorimeter.

Method of Procedure:

Place 250 c.c. of fourth normal sodium hydroxide, accurately measured in one graduated flask, into the (lower) calorimetric vessel. Put the covers, the brass stirrer and the Beckmann thermometer in position, after setting the mercury column so that it reads about half a degree from the bottom of the scale when placed in the alkali solution.

Pour about 260 c.c. of fourth normal hydrochloric acid into the upper vessel, then run it back into the flask.

Make up to exactly 250 c.c. In this way the error due to the acid adhering to the walls of the upper vessel when discharging the 250 c.c. into the calorimeter is eliminated.

Return the accurately measured acid to the upper vessel and place the ordinary thermometer in this solution.

See that both the acid and the base are, when placed in the upper and lower vessel, at approximately the same temperature.

Stirring both solutions, alternately read the two thermometers at 30-second intervals for 5 minutes. Estimate the thousandths of degrees on the Beckmann thermometer by means of a reading glass and the hundredths of degrees on the ordinary thermometer in the same way.

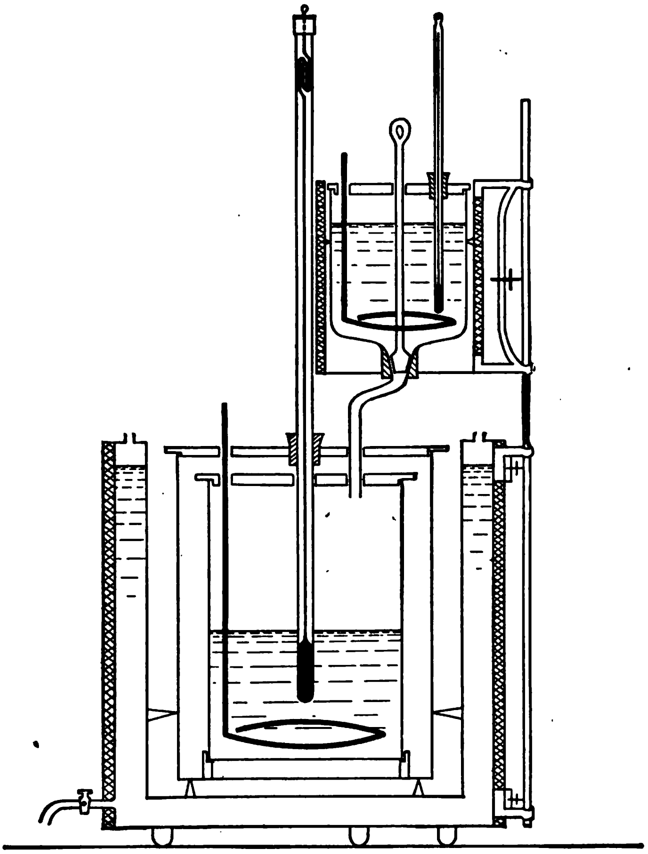

FIG. 18.

At the sixth minute pull the stopper and allow the acid to flow into the alkali. Stirring the mixture constantly, keep on taking readings every minute on the Beckmann thermometer for the next 10 minutes. Record these readings.

Finally compare both thermometers by placing the ordinary thermometer next to the Beckmann in the lower vessel and, while stirring constantly, take the readings on both alternately for at least three minutes.

In order to find the water equivalent of the calorimeter and stirrer, weigh the calorimeter and the brass stirrer on an ordinary balance (to tenths of a gram), both being thoroughly dried beforehand. See Table 8, Appendix, for specific heats.

The water equivalent of the portion of the Beckmann thermometer immersed in the liquid is readily found by water displacement. Counterpoise a tall narrow beaker of water on the ordinary balance by means of another beaker of water.

Carefully clamp the thermometer and support it on an iron stand so that only the bulb is immersed. The weight to be added on the other balance pan represents the volume of the water displaced by the glass and mercury of the bulb. Since 1 c.c. of glass has approximately the same water equivalent ($2.5 \times 0.19 = 0.47$) as 1 c.c. of mercury ($13.6 \times 0.034 = 0.46$) it is only necessary to multiply the weight of the displaced water by 0.47 to obtain the water equivalent of the bulb.

Since the stem of the thermometer above the bulb is also partly in the liquid, dip the thermometer as far in the water of the beaker as it was immersed in the solution in the calorimeter and again determine the weight of the water displaced.

Only one-fifth of the difference of this and the previous weight is solid glass (the stem being hollow); therefore take one-fifth of the difference of both weighings, multiply this by 0.47 and add this to the water equivalent of the bulb. This gives the total water equivalent of the immersed portion of the Beckmann thermometer.

Observations and Measurements:

	Readings on Beckmann thermometer (lower vessel)	Readings on ordinary thermometer (upper vessel)
At start	....	
After 30 seconds		....
1 minute	....	
90 seconds		....
2 minutes	....	
150 seconds		....
3 minutes	....	
210 seconds		....
4 minutes	....	
270 seconds		....
5 minutes	....	
6 minutes	....	
7	....	
8	....	
9	....	
10	....	
11	....	
12	....	
13	....	
14	....	
15	....	
Comparison of both thermometers (in lower vessel)		
	....	
		....
	....	
		....
	....	
		....

Weight of calorimeter ......g.

Weight of brass stirrer ....g.

Bulb of Beckmann thermometer immersed. Weight of water ....g.

Weight of water displaced when immersed to same depth as in calorimeter, ...g.

Calculations:

1. Plot temperatures as ordinates ($1° = 10$ cm.) against times as abscissae (1 min. $= 1$ cm.), recording *all* values in terms of the Beckman scale. By means of a straight line project the last seven or eight readings back to the perpendicular for the time of mixing and call the temperature corresponding to this intersection the true temperature attained on mixing.

2. Taking as t_B and t_A the increase in temperature of base and acid respectively, and assuming that the specific heat of the dilute acid and base is the same as that of pure water, namely 1 we have:

Heat absorbed by acid $= 250t_A$
Heat absorbed by base+calorimeter $= (250+W)t_B$
Heat liberated by neutralization $= 250t_A + 250t_B + Wt_B$, representing by W the water equivalent of calorimeter, stirrer and immersed portion of Beckmann thermometer.

3. Considering the fact that only 250 c.c. of a fourth normal base and acid were used, the result obtained above must be multiplied by 16 to obtain the molar heat of neutralization.

EXPERIMENT 18

HEAT OF COMBUSTION

Object:

To determine the heat of combustion of a given carbon compound.

References:

Textbooks:

Bigelow, XXVII, Thermochem. Data, p. 416
Getman, XI, Thermochemistry, p. 336
Jones, VI, Thermochemistry, p. 327
Lincoln, XXXIV, Thermochemistry, p. 418
Millard, VII, Thermochemistry, p. 198
Senter, VI, Thermochemistry, p. 145
Walker, XIII, Thermochem. Change, p. 133
Washburn, XIX, Thermochemistry, p. 242

Laboratory Manuals:

Ewell, IV, Thermochemistry, p. 193
Findlay, XIII, Thermochemistry, p. 285
Firth, XII, Thermochem. Meas., p. 79
Getman, VII, Calorimetry, p. 113
Gray, Heat of Combustion, p. 85
Pring, Part I, Calorimetry, p. 34
Roth (Cameron), IV, Thermochemistry, p. 65
Traube (Hardin), XV, Thermochemical Constants, p. 141

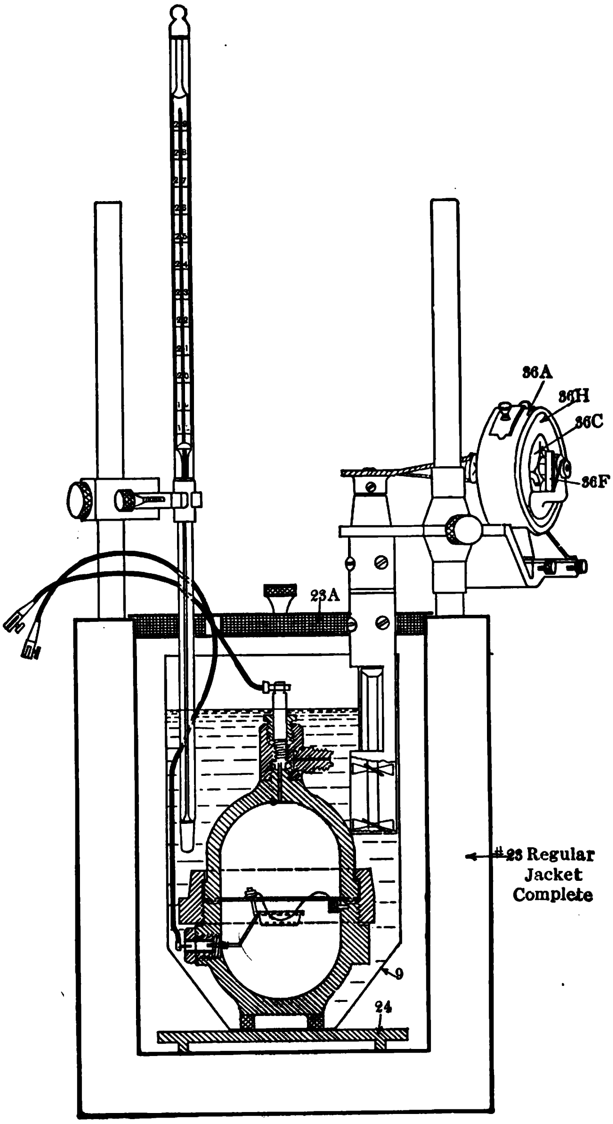

FIG. 19.

Apparatus and Chemicals required:

1 standard combustion bomb calorimeter (oxygen type) with accessories.[1]

Sample bottles containing pure cane sugar, benzoic acid and naphthalene for standardization purposes.[2]

Sample bottles containing organic compounds the heat of combustion of which is to be determined.

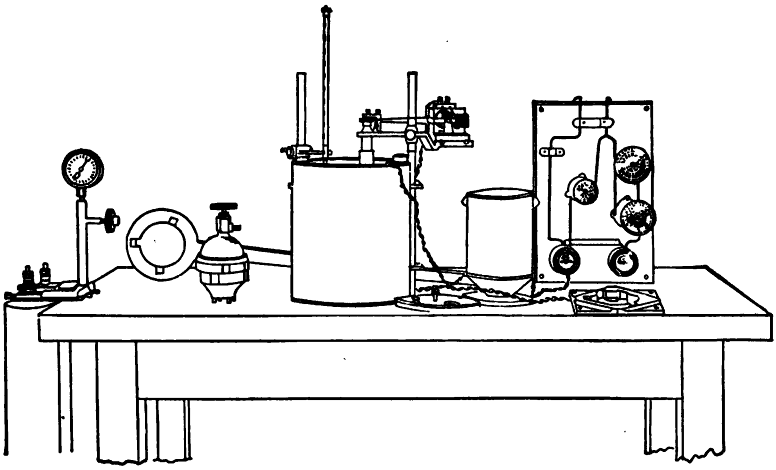

FIG. 20.

Fig. 19 and Fig. 20 show views of the Emerson bomb calorimeter which is widely used in this country for the determination of the heat value of coke, fuel oil, gasoline, etc.

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[1] In as much as complete calorimetric outfits are obtainable from dealers a minute catalogue of equipment is omitted. For careful work the adiabatic calorimeter equipped with vacuum walled jacket is preferable, but most laboratories would hesitate to use so expensive and fragile an apparatus with beginners. The directions for this experiment are therefore given for a non-adiabatic calorimeter and hold equally well for any of the well-known types of oxygen bomb.

[2] Obtainable from the Bureau of Standards, Washington, D. C. Complete details regarding the standardization of bomb calorimeters are found in Circular No. 11 of the Bureau of Standards.

Method of Procedure:

A. Determination of the "water equivalent" factor of the apparatus.

By means of a tabloid press, prepare a cartridge containing from 0.75 to 1.00 gram of benzoic acid, naphthalene or cane sugar.

Weigh this accurately and place it in the small crucible of the bomb. Place the crucible in its support, and attach a weighed iron fuse wire. This wire should be about 2 inches long and be coiled at the center, the coil being directly above the pellet in the crucible. Fasten the ends of the wire to the electrodes by wrapping them tightly. The fuse wire should dip down into, but not touch, the crucible.

Place the outside shell of the bomb in its holder. Carefully place the top in position on the bomb, then screw the two parts together tightly by means of the spanner.

Transfer the bomb to the oxygen fixture and connect the gauge stand to the bomb by screwing up the union. Use the wrench to secure a tight joint. Open the needle valve in the head of the bomb about two turns and carefully "crack" the valve leading from the oxygen cylinder. Do not permit the oxygen to rush from the cylinder or the gauge may be ruined.

When the pressure in the bomb has been increased to 20 atmospheres, close the valve in the cylinder and then the valve in the bomb.

Disconnect the bomb from the fixture and find out by carefully listening for the escape of gas whether there are any leaks.

If no leaks develop, place the bomb within the calorimeter, make the electrical connections and adjust the stirring device.

Using a 500-c.c. graduated flask, add sufficient water to immerse the bomb. (Most calorimeters require 2000 c.c.) It is of the greatest importance that the temperature of this water be about (but not more than) one degree Centigrade below room temperature.

Place the covers in position and clamp the thermometer (which should have a calibration certificate) with its bulb immersed at least 4 inches. In case a Beckmann thermometer is used it should be set so that the mercury thread stands near the bottom of the scale.

After the stirring outfit has been in operation for five or six minutes, take one-minute readings on the thermometer.

When 10 consecutive readings have been obtained, ignite the material in the bomb by closing the firing switch. This should be done exactly on the minute and a note made of the time of firing.

The temperature will rise rapidly during the next few minutes. Continue to take minute readings until at least 15 observations have been made after the maximum temperature is reached and the changes per minute are regular and very small.

When through with the run, remove the bomb, replace it in the holder, and gradually allow the products of combustion to escape through the valve. Unscrew the top of the bomb and note whether the sample was completely burned. Also see whether the iron wire was completely oxidized, or only partly. Any remaining pieces of iron may be collected and weighed and this weight subtracted from the weight of iron taken.

B. Determination of heat of combustion for an assigned carbon compound.

Prepare a tablet containing about 1 gram of the (solid) substance under investigation and weigh it accurately.

Follow the same direction as outlined under A and record the data obtained in the space left open for that purpose.

Carefully clean and dry the bomb before leaving the room.

Observations and Measurements:

A. Substance used: ...., weight ....g.

Thermometer used, No...... Room temperature, ...° C.

Volume of water used ....c.c., temperature of water, ...° C.

Weight of iron wire used: ....g.

Time	Temperature	Time	Temperature
....	....	....	....
....	....	....	....
....	....	....	....
....	....	....	....
....	....	....	....
....	....	....	....
....	....	....	....
....	....	....	....
....	....	....	....
....	....	....	....
....	....	....	....
....	....	....	....
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....	....	....	....
....	....	....	....
....	....	....	....
....	....	....	....

B. Substance used: ...., weight ....g.

Thermometer used, No. ..... Room temperature, ...° C.

Volume of water used ....c.c., temperature of water, ...° C.

Weight of iron wire used: ....g.

Time	Temperature	Time	Temperature
....	....	....	....
....	....	....	....
....	....	....	....
....	....	....	....
....	....	....	....
....	....	....	....
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....	....	....	....
....	....	....	....

Calculations:

1. Plot temperatures as ordinates versus times as abscissae on a sufficiently large scale. Extend the final radiation curve back to the ordinate for the time of firing by means of a straight line and call the temperature corresponding to this intersection the true temperature reached on combustion. The distance from this temperature to that attained at the moment of firing gives the corrected rise in temperature.

2. Knowing the heat of combustion of the substance used (benzoic acid, 6320 cal./gm.; cane sugar 3950 cal./gm.; naphthalene 9693 cal./gm. and iron 1600 cal./gm.) calculate the heat liberated by the combustion. This heat divided by the corrected rise in temperature gives the apparent weight of water of the whole system which, minus the weight of water introduced gives the " water equivalent " of the apparatus.

3. Using the value for the water equivalent found under 2 calculate the heat of combustion for the assigned substance in calories per gram and the molecular heat of combustion in Calories per mole.

EXPERIMENT 19

HEAT OF SOLUTION

Object:

To determine the heat of solution of a given salt.

References:

Textbooks:

Bigelow, XXVII, Thermochem. Data, p. 413
Getman, XI, Thermochemistry, p. 343
Lincoln, XXXIV, Thermochemistry, p. 427
Millard, VII, Thermochemistry, p. 206
Morgan, VII, Thermochemistry, p. 216
Walker, XIII, Thermochem. Change, p. 137
Washburn, XIV, Solutions, IV, p. 175

Laboratory Manuals:

Briggs, XVII, Thermochemistry, p. 82
Ewell, IV, Thermochemistry, p. 187
Findlay, XIII, Thermochemistry, p. 283
Firth, XII, Thermochem. Meas., p. 78
Getman, VII, Calorimetry, p. 109
Gray, Ex. 19, Heat of Solution, p. 82
Traube (Hardin), XV, Thermochem. Constants, p. 137

Journal Article:

Lamb and Simmons, Jour. Am. Chem., Soc. *43*, p. 2190 (1921)

Apparatus and Chemicals required:

1 vacuum cylinder (suitable dimensions 7 cm. × 30 cm., contents 1000 c.c.)
1 Beckmann thermometer
1 thermometer (0–100°)
1 cardboard jacket and a perforated cork to protect vacuum cylinder.
1 reading glass
1 glass stirrer with pulley wheel attached
1 small motor with belt
1 heating coil wrapped around U-tube with platinum leads ending in mercury cups (see Fig. 21). The wire should preferably have a negligible temperature coefficient (manganin). Resistance should be about 5 ohms. Coil should be shellacked.
1 ammeter, 0–1.5 amperes, graduated to 0.01 amp.
1 six-volt, lead storage battery
1 switch
10 feet of No. 18 annunciator wire
1 large iron stand
2 iron rings
3 small iron clamps
1 thin-walled test tube
1 250-c.c. graduated flask
Sample bottles with pure finely powered salts.

The assembled apparatus is shown in Fig. 21. The Dewar flask is wrapped in cardboard and held securely on the base of the iron stand by means of two iron rings. The glass stirrer operates near the bottom of the cylinder. The water equivalent of the whole system is determined by means of the electric heating device. Since the coil is in intimate contact with the water its resistance does not materially change during a determination. Knowing the resistance of the coil and the current input as measured by the ammeter the amount of heat dissipated within the Dewar flask can be calculated.

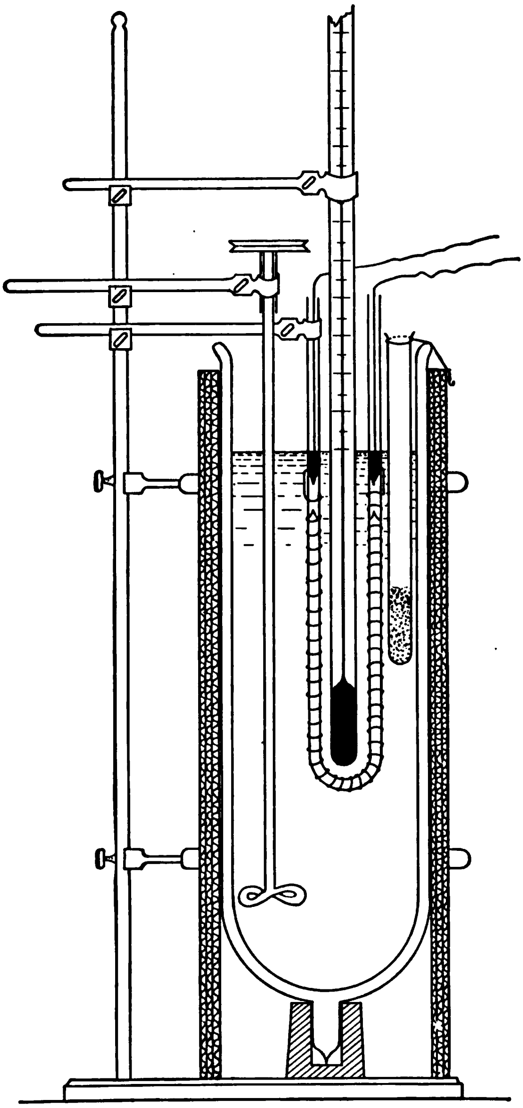

FIG. 21.

Method of Procedure:

Assemble the apparatus, observing care that the stirrer operates freely and does not strike the walls of the cylinder. Fill the Dewar cylinder with 750 c.c. of water measured out in the graduated flask. The temperature of the water should be about one degree (not more!) below room temperature.

Weigh out sufficient finely powdered salt (assigned for the experiment) to give 1 mole of salt in 200 moles of water, place this amount in the thin-walled test tube and suspend the latter in the water of the vacuum cylinder.

Clamp the Beckmann thermometer in place, after having " set " the mercury thread so that it stands midway on the scale when the bulb is immersed in the water.

Start the stirrer and test the electrical connections by closing the switch for an instant. The current should be equivalent to an expenditure of about 5 watts.

After about 5 minutes commence to record one-minute readings of temperature, and continue until 5 readings of constant difference have been obtained. On the next full minute close the heating switch and record half-minute ammeter readings and one-minute temperature readings.

Allow the current to pass until the temperature has risen about one degree, stop on the full minute, and remove the copper leads from the mercury cups.

After the current has been interrupted continue the temperature readings for ten more minutes.

At the end of this period quickly lift the test tube out of the water and shake the salt into the cylinder, noting the exact time at which the transfer was made.

Replace the empty tube immediately and continue the one-minute temperature readings for at least ten minutes.

Stop the motor and remove the heating coil from the solution.

Observations and Measurements:

Volume of water used......c.c. Temperature ....°
Room temperature ....°
Salt used ...., weight of salt ....g.
Resistance of heating coil ....ohms.

Time	Temperature readings	Current readings
....	....	....
....	....	....
....	....	....
....	....	....
....	....	....
....	....	....
....	....	....
....	....	....
....	....	....
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....	....	....
....	....	....
....	....	....
....	....	....
....	....	....
....	....	....

Calculations:

1. Calculate the electrical energy expended in the heating coil and from this determine the number of calories produced.

2. Plot the time-temperature curve on a sufficiently large scale and determine the true rise in temperature caused by the electrical heating. From this rise find the water equivalent of the system.

3. Plot the time temperature curve for the period before and after adding the salt to the water and determine the true change in temperature caused by the solution of the salt. Knowing the water equivalent find the heat of solution of the salt used.

EXPERIMENT 20

ADSORPTION OF DISSOLVED SUBSTANCES BY CHARCOAL

Object:

To study the adsorption of acetic acid by charcoal and to test the validity of the adsorption equation:

$$x = k \cdot y^n$$

References:

Textbooks:

Bigelow, XVIII, Solutions, III, p. 241
Getman, X, Colloids, p. 320
Lincoln, XXXV, Colloid Chem., p. 454
Millard, IV, Solid Solutions, p. 107
Senter, XII, Coll. Soln's: Adsorption, p. 324
Washburn, XXV, Disperse Systems, p. 364

Laboratory Manual:

Briggs, XVI, Colloid Chemistry, p. 73

Special Texts:

Arrhenius, Theories of Solutions, Lect. 4, Phen. of Ads., p. 55
Bancroft, Applied Colloid Chemistry, III, Ads. from Solution, p. 100
Hatschek, Lab. Manual of Elem. Colloid Chemistry, XVIII, Determ'n of an Ads. Isotherm, p. 116
Ostwald and Wolski, Prakticum der Kolloidchemie, VII, Adsorption, p. 104
Willows and Hatschek, Surface Tension and Surface Energy, V, p. 52

Apparatus and Chemicals required:

1 iron stand
1 burette holder
2 50-c.c. burettes
1 pipette, calibrated to deliver 10 c.c.
1 filter stand
2 funnels, 2.5 inch
6 125-c.c. Erlenmeyer flasks, cork-stoppered (or 6 glass-stoppered, 125-c.c. iodine flasks)
6 125-c.c. wide-mouth Erlenmeyer flasks
2 100-c.c. beakers
1 bottle containing a suitable, high-grade charcoal
1 stock bottle containing approximately 0.4 N acetic acid (23 c.c. of glacial acetic acid made up to 1 liter)
1 stock bottle containing 0.1 N alkali
1 small bottle containing phenolphthalein indicator
1 wash bottle containing distilled water
Filter paper (diameter 8 cm.)

Method of Procedure:

Into each of the 6 stoppered Erlenmeyer flasks, labeled 1–6 (thoroughly cleaned and dried beforehand), place 5 grams of charcoal,[1] accurately weighed on an analytical balance.

Into flask No. 6 run 48 c.c. of the 0.4 N, acetic acid, measured accurately from the 50-c.c. burette. In No. 5 place 24 c.c. of acid; in No. 4, 12 c.c.; in No. 3, 6 c.c.; in No. 2, 3 c.c.; and finally in No. 1, 1.5 c.c.

Rinse the burette twice with distilled water, fill it to the zero mark with distilled water and add to flask No. 5, 24 c.c. of water; to No. 4, 36 c.c.; to No. 3, 42 c.c.; to No. 2, 45 c.c.; and finally to No. 1, 46.5 c.c. of water. All these different volumes should be measured very carefully and added slowly so as to allow the liquid in the burette time to drain.

---

[1] Inasmuch as the different forms of charcoal show considerable variation in adsorbing power, it is necessary that preliminary experiments be conducted by the instructor to determine the proper amount of charcoal to be used. Blood charcoal is recommended as giving the most consistent results, but even with such an apparently uniform substance it is not always certain that equal weights represent equal surfaces. Great care should be taken therefore in selecting a high-grade charcoal. The proper amount to be used, which may be either more or less than 5 grams, is established beforehand.

Stopper the flasks and shake the same successively, starting with No. 6, for at least 10 minutes.

Pipette off 10 c.c. of the stock solution of acid, place the same in a 100-c.c. beaker (allowing the pipette to drain for 25 seconds; this same time limit should be observed in all the subsequent operations in taking 10-c.c. samples), add a few drops of phenolphthalein and titrate with the tenth normal alkali in the second burette. The end point is reached when a faint pink color persists, on shaking. Always titrate to this same pink color. Repeat the titration twice. The determinations should check to within 0.1 c.c.

Filter the contents of flask No. 6 into a dry and clean wide-mouth Erlenmeyer flask (also labeled 6) and titrate 2 (or 3) 10-c.c. samples (checking to 0.1 c.c.) as directed above.

Filter the contents of flask No. 5 next and take 2 (or 3) 10-c.c. samples. Titrate as before.

From the filtrates of flasks No. 4 and No. 3 withdraw two 20-c.c. samples (using the 10-c.c. pipette) and titrate as before.

From the filtrates of flasks No. 2 and No. 1 withdraw one 40-c.c. sample and titrate this sample very carefully, as only very little alkali will be required, and not enough solution is left to repeat the titration.

Observations and Measurements:

Charcoal used taken from bottle labeled ....

Quantity used .... grams. Volume of liquid, 48 c.c.

Titrations:

	Stock bottle	No. 6	No. 5	No. 4	No. 3	No. 2	No. 1
	......	......	......	......	......	......	......c.c.
	......	......	......	......	......		
	......	......	......				
Average	......	......	......	......	......		

Multiply the averages obtained for the stock bottle solution and Nos. 6 and 5 by 4.8, for Nos. 4 and 3 by 2.4 and for Nos. 2 and 1 by 1.2.

Calculations:

1. Tabulate the results obtained above in the following table:

	$x+y$ Initial concentration in c.c. of 0.1 N alk. for 48 c.c. of sol.	y Concentration of solution at equilibrium in c.c. of 0.1 N alk. for 48 c.c. of solution	x Amount adsorbed expressed in c.c. of 0.1 N alk.
No. 1	......(1/32 of 6)	......	......
No. 2	......(1/16 of 6)	......	......
No. 3	......(1/8 of 6)	......	......
No. 4	......(1/4 of 6)	......	......
No. 5	......(1/2 of 6)	......	......
No. 6	......	......	......

2. Plot the data obtained from columns 2 and 3 on cross-section paper (choosing proper units); the equilibrium concentrations (y) as abscissae against the amounts absorbed (x).

3. Calculate the values for log y and log x and plot these values on a sufficiently large scale, the logs y as ordinates and the logs x as abscissae. In case the adsorption equation $x = k.y^n$ holds, the 6 points will lie approximately on a straight line. Draw the straight line joining these points and derive from this line the values for n and k. Substitute the values thus obtained in the equation and calculate for each value of y the corresponding value of x. Tabulate the results in the following table:

	log y	log x	x observed	x calculated
No. 1	......	......	......	......
No. 2	......	......	......	......
No. 3	......	......	......	......
No. 4	......	......	......	......
No. 5	......	......	......	......
No. 6	......	......	......	......

EXPERIMENT 21

CONDUCTIVITY AND DEGREE OF IONIZATION

Object:

To determine degree of dissociation by the conductivity method, and to verify Ostwald's dilution law for weak electrolytes.

References:

Text books:

Bigelow, XXVIII, The Quantity Factor, p. 437
Getman, XV, Electr. Conductance, p. 471
Jones, VII, Electrochem., p. 377
Lincoln, XXVII, Electr. Conductance, p. 311
Millard, VI, Soln's of Ionized Subs., p. 164
Morgan, IX, Electrochem., p. 396
Senter, X, Electr. Conductance, p. 254
Walker, XXII, Electrolytes & Electrolysis, p. 243
Washburn, XVII, Conductance & Degree of Ionization, p. 212

Laboratory Manuals:

Briggs, VIII, Dissociation, p. 29
Ewell, VIII, Electrochem., p. 279
Findlay, IX, Cond. of Electrolytes, p. 165
Firth, XIV, Electr. Cond., p. 91
Getman, X, Conductivity, p. 159
Gray, Cond. of Electrolytes, p. 139
Pring, Pt. 1, Electrochem., p. 72
Roth (Cameron), IX, Electr. Cond., p. 127
Traube (Hardin), VI, Electr. Cond. of Liquids, p. 58

Apparatus and Chemicals required:

1 thermostat, capable of regulation to within 0.1°
1 conductivity cell, either the well-known Arrhenius cell or a simple immersion type with so-called dipping electrodes
1 Kohlrausch conductivity bridge or slide wire bridge
1 resistance box
1 small, high-frequency induction coil
1 telephone receiver
1 battery of 2 dry cells
20 feet of insulated copper wire
1 10-c.c. pipette, calibrated to deliver
1 10-c.c. pipette, calibrated to contain
1 bottle with fiftieth-normal potassium chloride
1 bottle with CO_2-free distilled water
1 bottle with sixteenth-molar solution of acetic, benzoic, succinic or other weak acid
1 small bottle of mercury

Fig. 22 illustrates the manner of setting up and connecting the apparatus. The conductivity cell with its platinum electrodes well coated with platinum black is clamped to the thermostat in such a way that it is immersed up to within 1 inch of the top. It is connected in the right arm of the bridge and the known resistance in the left arm. The induction coil is connected across the ends of the bridge and the telephone from between the known resistance and the conductivity cell, to the point of contact on the bridge wire. With this scheme of wiring, the resistance (x) of the solution in the cell is given by the expression $x=\frac{R\cdot b}{a}$ and the observed conductivity $\left(c=\frac{1}{x}\right)$ by $c=\frac{a}{R\cdot b}$.

Method of Procedure:

It is important that the position of the electrodes be not altered during the course of the experiment; nor must the platinum black surfaces of the electrodes be touched so as to change their character in any way.

Pour out any liquid which may be in the cell and rinse it, together with the electrodes, three times with fiftieth-normal KCl solution, using a few c.c. at a time. The electrodes are best rinsed by gently moving them up and down in the cell a few times.

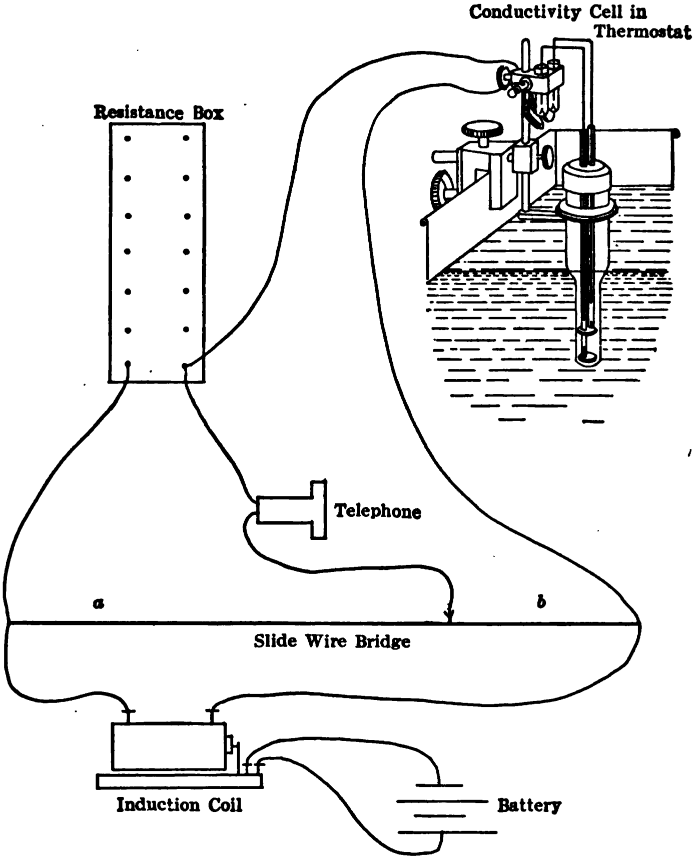

FIG. 22.

After thorough washing, introduce 10 c.c. of the fiftieth normal KCl solution, place the cell in the thermostat and determine the conductance of the solution at 25°. See that a minimum on the telephone is obtained with the point of contact near the middle of the bridge wire. Since temperature changes have a marked effect upon conductivity the temperature should not be permitted to differ more than 0.1° from 25°.

The value of the cell constant is obtained by dividing the specific conductance of fiftieth-normal potassium chloride at 25° (see Table 9) by the observed conductivity as determined above.

Remove the potassium chloride solution, rinse the cell and electrodes with distilled water and dry them.

By means of the 10-c.c. pipette calibrated *to deliver*, introduce 10 c.c. of the sixteenth-molar acid assigned for observation and determine its conductivity at 25°.

Add to the solution in the cell by means of the 10-c.c. pipette calibrated *to deliver* 10 c.c. of CO_2-free distilled water; thoroughly stir the resulting mixture by moving the electrodes gently up and down; then withdraw 10 c.c. by means of the 10-c.c. pipette calibrated *to contain* and discard the portion taken out.

Measure the conductivity of the thirty-second molar solution of acid thus obtained.

Proceeding as above, dilute the solution in the cell progressively to M/64, M/128, M/256 and M/512 and determine the conductivity each time.

When finishing the experiment, either leave the last solution in the cell or fill it with distilled water.

Do not leave the cell dry.

Observations and Measurements:

Determination of cell constant using N/50 KCl.
Bridge reading, left ..... right ..... R = ... ohms
Assigned acid: ....

Bridge reading	M/16	M/32	M/64	M/128	M/256	M/512
Left:	....	....	....	....	....	....
Right:	....	....	....	....	....	....
Resistance (R)	....	....	....	....	....	....

Calculations:

1. Calculate the cell constant.

2. Compute the conductance, specific conductance and equivalent conductance for each dilution of acid.

3. From Table 10 obtain the value of the equivalent conductance at infinite dilution of the acid used and then calculate the degree of ionization at each concentration.

4. Calculate the constant of Ostwald's dilution law for each dilution.

5. Plot specific and equivalent conductances, also the degrees of ionization against volumes as abscissae.

6. Tabulate the values found in the following table:

Assigned weak acid ....

	M/16	M/32	M/64	M/128	M/256	M/512
Obs. conductance:	....	....	....	....	....	....
Spec. conductance	....	....	....	....	....	....
Equiv. conductance:	....	....	....	....	....	....
Degree of ioniz.	....	....	....	....	....	....
Ionization const.	....	....	....	....	....	....

EXPERIMENT 22

TRANSPORT NUMBERS[1]

Object:

To determine the transport numbers of the ions Ag^+ and NO_3^-.

References:

Textbooks:

Bigelow, XXVIII, The Quant. Factor, p. 442
Getman, XVIII, Electr. Cond., p.
Jones, VII, Electrochem., p. 368
Lincoln, XXVII, Electr. Cond., p. 308
Millard, VI, Soln's of Ion. Subs., p. 172
Morgan, IX, Electrochem., p. 388
Senter, X, Electr. Cond., p. 246
Walker, XXII, Electrolytes & Electrolysis, p. 237
Washburn, XVIII, Electr. Trans., p. 229

Laboratory Manuals:

Ewell, VIII, Electrochem., p. 292
Findlay, X, Transport Nos., p. 206
Firth, XIII, Determ'n of Trans. Nos., p. 87
Getman, XII, Meas. of Trans. Nos., p. 212
Gray, Ex. 31, Determ'n of Trans. Nos., p. 129
Roth (Cameron), X, Faraday's Law & Trans. Nos., p. 146

Special Texts:

Allmand, Applied Electrochemistry, V, Ionic Transport, p. 52
LeBlanc (Whitney-Brown) Textbook of Electro-chemistry, IV, Migration of Ions, p. 71

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[1] The minimum time required for the completion of this experiment is about 3 hours.

Apparatus and Chemicals required:

1 transport number cell with silver electrodes
1 copper coulometer
1 ammeter (0–1 ampere, graduated in hundredths of amperes)
1 knife switch
1 thirty-two volt battery, or direct current source of greater potential
1 adjustable resistance (maximum resistance 200 ohms)
2 iron stands
2 clamps
4 screw connectors
8 feet of flexible insulated copper wire
1 burette
1 burette holder
2 250-c.c. stoppered Erlenmeyer flasks, marked *A* and *M*
2 250-c.c. Erlenmeyer flasks
1 pair of crucible tongs
1 25-c.c. pipette
1 50-c.c. graduated cylinder
1 bottle containing copper coulometer solution (150 grams of crystallized copper sulphate $CuSO_4 \cdot 5H_2O$ dissolved in a mixture of 1000 c.c. of distilled water, 28 c.c. of H_2SO_4, specific gravity 1.84, and 60 c.c. of ethyl alcohol)
1 stock bottle containing silver nitrate solution, approximately 8.5 grams per liter
1 stock bottle containing standard KCNS solution (1 c.c. being equivalent to 0.005 gram of silver)
1 100-c.c. bottle containing a saturated solution of ferric ammonium sulphate, to which sufficient nitric acid has been added to give the solution a clear pale yellow color (this is to suppress hydrolysis)
1 wash-bottle containing distilled water
1 bottle of alcohol

The apparatus is shown (partly schematically) in Fig. 23. A three-compartment cell as given in the sketch is preferable for the use of beginners, as it offers less opportunity for the introduction of errors in drawing off the contents of the various compartments at the end of the run. A simple and efficient copper coulometer may be made from a small crystallizing dish about 2 inches in diameter, and 2 square sheets of copper, held in position by brass spring clips attached to a hardwood electrode holder, which rests on the dish, as shown in Fig. 24. This

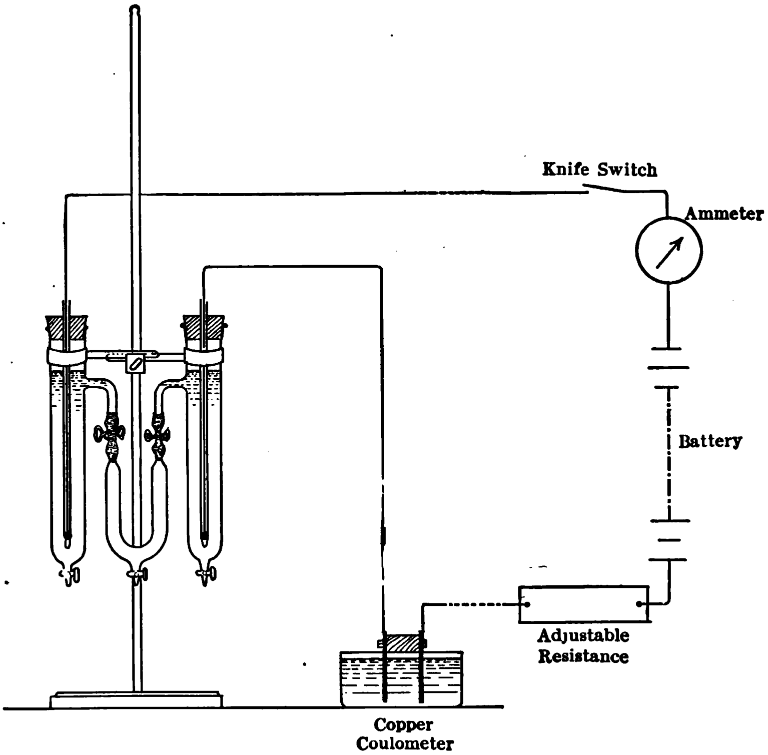


FIG. 23.

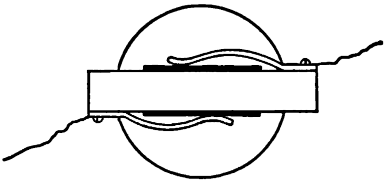


FIG. 24.

construction provides a convenient means of holding the electrodes and does away with the necessity of terminals soldered to the copper sheets. With the three-compartment cell a potential of 32 volts is required to furnish the necessary current.

Method of Procedure:

Wash the coulometer cathode with distilled water, then with alcohol. Grasp it with the crucible tongs, and ignite the alcohol adhering to it. The cathode will be warmed sufficiently by the burning alcohol to dry it completely. When it is cool, weigh it accurately to a tenth of a milligram.

After weighing the cathode, clean and dry the two stoppered flasks, marked *A* and *M*, and weigh the one marked *A* to 0.01 gm.

While the first student is carrying out the operations directed above, the second should clean and dry the cell, fill it with stock silver nitrate solution, and assemble the apparatus, observing care that the electrical connections are made as per the diagram.

Nearly fill the coulometer with coulometer solution, and adjust *R* to give its maximum resistance.

Close the switch, noting the time, and adjust the resistance to give a current of between 0.01 and 0.02 ampere. The duration of passage of current should be two hours. During this time, the strength of the current should be held constant.

At the conclusion of the electrolysis, interrupt the current, noting the time, carefully close the pinch-cocks, and withdraw the contents of the anode compartment into stoppered Erlenmeyer marked *A*, rinsing the compartment and the anode with a small amount of the original solution from the $AgNO_3$ stock bottle. Open the pinch-cock next to the anode compartment, and withdraw the contents of the middle compartment into the flask marked *M*, but do not rinse this compartment.

Remove the cathode from the coulometer, rinse it with water, then alcohol, and dry and weigh as before.

Weigh flask *A*, with its contents, to 0.01 gm.

While the first student is weighing the flask and cathode, the second should determine the concentrations of silver in the $AgNO_3$ solution in the stock bottle, and in the solution in flask *M*, by titration with standard KCNS, as follows:

Rinse the 25-c.c. pipette with a small portion of the solution to be analyzed, discard this portion, then withdraw a 25-c.c. sample of the solution, run it into a 250-c.c. Erlenmeyer flask, add 50 c.c. of distilled water, and 1 c.c. of ferric ammonium sulphate solution, then run standard KCNS in from the burette until the first permanent red tint is obtained. Check the result by a second titration.

If the concentrations of Ag$^+$ in the stock bottle and in the solution from the middle compartment do not agree, diffusion has taken place, and the results will be inaccurate.

After weighing, analyze the solution in flask *A* in like manner.

Before leaving the apparatus, see that the clamps and stopcocks are open, so that the cell will drain.

Pour the coulometer solution back into the proper stock bottle.

Observations and Measurements:

1 c.c. standard thiocyanate solution equivalent to ....gms. Ag$^+$
Time of starting electrolysis ....
Time of stopping electrolysis ....
Current strength used ....
Weight of copper cathode after electrolysis ....
Weight of copper cathode before electrolysis ....
Cathode gain ....
Weight flask *A* plus solution ....
Weight flask *A* ....
Weight anode solution ....
Titrations: c.c.
Stock solution ....
....
Ave. ....
Middle compartment ....
....
Ave. ....
Anode compartment ....
....
Ave. ....

Calculations:

1. From the weight of copper deposited in the coulometer, calculate the weight of silver dissolved from the anode.

2. From the total weight, and the analysis, of the anode solution, calculate the amount of Ag^+ actually found in the anode compartment, assuming that the density of the dilute solution is unity.

3. From the analysis of the stock solution calculate the quantity of Ag^+ originally present in the anolyte, assuming as before that its density is unity.

4. From the data obtained in 1, 2, and 3, calculate the transference numbers of the two ions.

EXPERIMENT 23

HYDROGEN ION CONCENTRATION: ELECTROMETRIC TITRATION

Object:

To measure hydrogen ion concentration electrometrically, to employ these measurements in the titration of acids and bases, and to observe the point of color change of some indicators.

References:

Textbooks:

Lincoln, XXXVII, Electromotive Force, p. 540
Millard, XVI, Electrochemistry, p. 391

Special Texts:

Clark, Determination of Hydrogen Ions, which contains a fairly complete bibliography up to 1920.
Leach-Winton, Food Inspection and Analysis, p. 1021.
Lewis, A System of Physical Chemistry, Vol. 1., pp. 252–261.

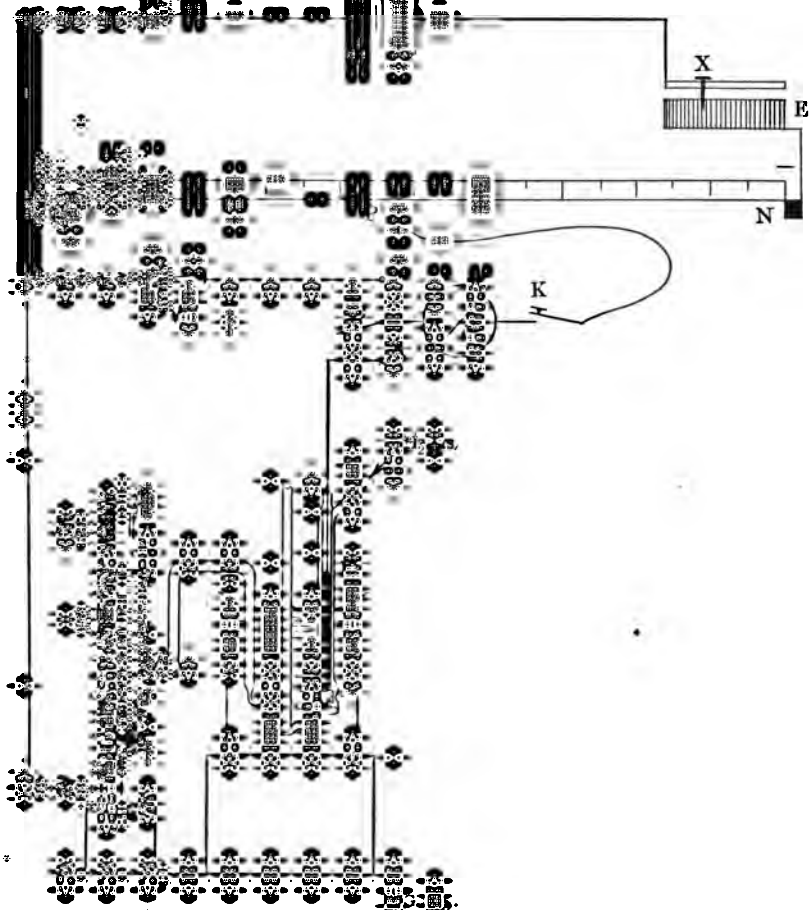
Apparatus and Chemicals required:

1 Weston standard cell
1 calomel electrode (normal KCl)
1 hydrogen electrode, preferably of the Hildebrand type
1 slide wire potentiometer, resistance about 3 ohms.
1 battery, approximately 2 volts
1 adjustable resistance of about 10 ohms maximum
1 single-pole, single-throw switch

1 single-pole, double-throw switch
1 portable galvanometer, sensitive to 2 micro-amperes
1 key
1 sliding contact
1 hydrogen tank or Kipp generator
2 gas wash bottles containing strong alkali and carefully prepared alkaline pyrogallol respectively
5 feet 4 mm. rubber tubing
1 hydrogen control valve
1 motor and stirring device
1 iron stand
5 iron clamps
2 burettes graduated to 0.05 c.c.
1 25-c.c. pipette
1 150-c.c. beaker
15 feet, No. 20 insulated copper wire
1 25-c.c. graduated cylinder
4 stock bottles with tenth-normal HCl, CH_3COOH, NaOH and NH_4OH respectively
3 indicator solutions (methyl orange, phenolphthalein and cochineal)

The arrangement of the apparatus is shown in Fig. 25. The battery *B* supplying current to the potentiometer wire may consist of two dry cells, although it will be found much more satisfactory to use two Edison cells or one lead accumulator. In the circuit with the battery and the potentiometer wire *MN* are the switch *S* and the adjustable resistance *E*. The calomel electrode *C* and the positive side of the standard cell *W* are connected to the positive end *M* of the potentiometer wire. The negative side of the standard cell and the hydrogen electrode *H* are connected to the outside contacts of the double-throw switch *D*, while one terminal of the galvanometer *G* is attached to the central terminal of the switch *D*. The other end of the galvanometer is connected through the key *K* to the sliding contact *P*.

The iron stand with clamps carrying the motor, stirring [illegible] electrode and burettes is [illegible] preferably the hydrogen [illegible] out. In case a generator [illegible] d be employed.



[illegible]

[illegible]

[illegible] orce measurements with [illegible] o establish a known drop [illegible] ortion of the wire. The [illegible] ell is 1.0186 volt at 20°

(temperature coefficient between 15 and 25° equals −0.0004 per degree). If therefore, the sliding contact be set at 50.93 cm. (for a room temperature of 20°) and the current in the potentiometer circuit so regulated that the drop in potential along the wire between *M* and *P* is equal to the potential of the standard cell, then each centimeter on the potentiometer scale from *M* to any point *p* will correspond to a potential of 0.02 volt.

In order to regulate the current in the potentiometer circuit, throw switch *D* into position *a*, thereby making connection with the standard cell. Close *S* and set *P* at the proper reading (50.93), then close key *K* for an *instant* by a *light tap* and note which way the galvanometer needle is deflected. Do not allow an appreciable current to flow through the standard cell or its E.M.F. will be altered. Shift contact point *X* and again close the galvanometer key *K*. A few observations of this character will suffice to adjust *X* until no deflection is noted when *K* is closed. When this is the case, the drop along *MP* is equal to the E.M.F. of the standard cell.

Having properly adjusted the current of the potentiometer circuit, proceed with the measurement of the electromotive force set up when the side arm of the calomel electrode and the hydrogen electrode are immersed in a solution containing hydrogen ions.

A. Titration of NaOH with HCl.

Slip the wooden block from beneath the titration beaker, empty the latter and rinse with distilled water, taking care to rinse the electrodes and stirring device also.

Replace the beaker, add 25 c.c. of distilled water, pipette 25 c.c. of tenth normal NaOH into it and add two drops of methyl orange. In rinsing the beaker and the electrodes, work rapidly, in order that the hydrogen electrode may not be exposed to the air longer than is absolutely necessary. Fill one burette with tenth normal HCl.

Start the stirring device and pass a brisk stream of hydrogen through the electrode.[1]

[1] See Clark, l.c., p. 133.

Throw switch *D* into position *b*, thereby connecting the hydrogen electrode with the sliding contact *P*. Depress *K* for an instant and adjust the position of *P* until no deflection is noted on the galvanometer when the key is closed. Avoid passing a large current through the " gas chain " or it will become polarized.

Record the position of *P*, taking consecutive half-minute readings until a constant value is obtained.

Add 5 c.c. of HCl from the burette and obtain a new value. Continue the addition of acid, measuring potentials after adding 10, 15, 18, 21, 22.5, 23, 23.6, 24, 24.2, 24.3, ... , ... 26.4, 26.5, 27, 27.5, 28, 29 and 30 c.c. Make note of first and second color change of the indicator.

B. Titration of NH_4OH with HCl.

Check the adjustment of the potentiometer current by throwing the switch *D* into position *a*, setting the movable contact at 50.93 and if necessary readjusting the position of the contact *X* as previously directed.

After rinsing the titration vessel, electrodes and stirrer, place 25 c.c. of distilled water and 25 c.c., accurately measured, of tenth normal ammonia solution in the beaker. Add two drops of cochineal indicator.

Proceed as under A, recording simultaneously volumes of acid added, and potentiometer readings. Note the exact point at which the indicator changes.

C. Titration of Acetic Acid with NaOH.

Check the potentiometer current, replace the solution in the beaker with 25 c.c. of distilled water and 25 c.c. of acetic acid.

After adding two drops of phenolphthalein add, in this case from the second burette, tenth normal NaOH.

Record volumes and potentiometer readings as before and note the volume when the first permanent faint pink appears in the beaker.

Observations and Measurements:

A NaOH+HCl Methyl orange c.c. pot. E.M.F. *pH* read.	B NH_4OH+HCl Cochineal c.c. pot. E.M.F. *pH* read.	C CH_3COOH+NaOH Phenolphthalein c.c. pot. E.M.F. *pH* read.
....	....	....
....	....	....
....	....	....
....	....	....
....	....	....
....	....	....
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....	....	....
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....	....	....
....	....	....
....	....	....
....	....	....
....	....	....
....	....	....
....	....	....
....	....	....
....	....	....

Calculations:

1. Multiply the potentiometer readings by 0.02 in order to obtain the E.M.F.

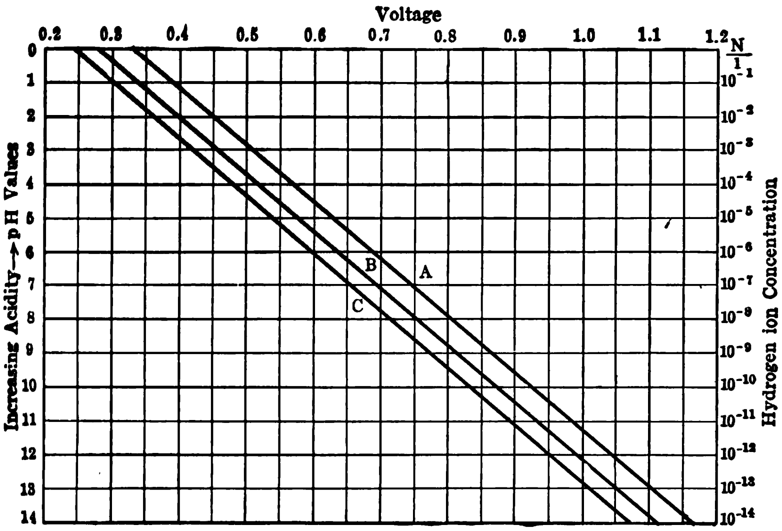
2. Calculate the value of *pH* in each case from the equation for the cell: [calomel electrode (containing normal KCl) || solution || hydrogen electrode]

$$\text{E.M.F.} = 0.283 + 0.0591\ pH$$

or, approximate the value of *pH* from the *pH*-E.M.F. curve (Fig. 26).

3. Plot the number of c.c. of reagent added against *pH* for each titration made, noting the neutral point ($pH=7$) and also the point at which the indicator changed color.

4. If the latter did not change at the "neutral point" explain the discrepancy.



Graphs showing relation between voltage of gas chain and hydrogen ion concentration (pH).

A. for a 0.1 Normal KCl calomel electrode.

B. for a Normal calomel electrode.

C. for a saturated KCl calomel electrode.

FIG. 26.

EXPERIMENT 24

ELECTROMOTIVE FORCE MEASUREMENTS: CONCENTRATION CELLS

Object:

To measure the electromotive force of concentration cells, to determine the concentration of a $AgNO_3$-solution, and to measure the solubility of AgCl.

References:

Textbooks:

Bigelow, XXIX, The Intensity Factor, p. 476
Getman, XVII, Electromotive Force, p. 549
Jones, VII, Electrochemistry, p. 442
Lincoln, XXXVII, Electromotive Force, p. 526
Millard, XVI, Electrochemistry, p. 371
Morgan, IX, Electrochemistry, p. 422
Senter, XIV, Electromotive Force, p. 367
Walker, XXX, Electromotive Force, p. 363
Washburn, XVI, Conduction of Electricity, p. 201

Laboratory Manuals:

Ewell, Introduction, p. 57
Findlay, XI, Meas. of E.M.F., p. 213
Firth, XV, Meas. of E.M.F., p. 107
Getman, XI, Electromotive Force, p. 183
Gray, Electromotive Force, p. 159
Pring, Pt. I, Electrochem., p. 81
Roth (Cameron), II, Meas. of Diff. of Pot., p. 152

Apparatus and Chemicals required:

1 potentiometer, accurate to ±0.5 millivolt[1]
1 adjustable resistance for potentiometer circuit, maximum resistance 1000 ohms
1 portable galvanometer, sensitive to 0.1 microampere
1 standard cell
1 double pole, double-throw switch
2 single-contact keys
1 10,000-ohm (approximate) resistance coil, used as protective resistance
1 knife switch
1 storage cell
5 " half-cell " elements, with silver electrodes
1 50-c.c. beaker
1 100-c.c. beaker
5 small stands for supporting half-cells
4 small screw connectors
20 feet, insulated copper wire
1 bottle containing normal $AgNO_3$
1 bottle containing tenth-normal $AgNO_3$
1 bottle containing hundredth-normal $AgNO_3$
bottles containing $AgNO_3$ of varying concentration (between normal and hundredth-normal) normality to be determined by students
1 bottle containing tenth-normal KCl solution
1 bottle containing normal-KNO_3 solution
1 bottle for silver residues

The concentration cell is shown in Fig. 27, and the wiring diagram in Fig. 28. A student type potentiometer will be found to be sufficiently accurate for the needs of this exercise, and less liable to injury in the hands of beginners than the more expensive instruments. With this potentiometer, it is advisable that a three-dial resistance box be used for the adjustable resistance *R*, although an adjustable sliding contact tube resistance will do just as well.

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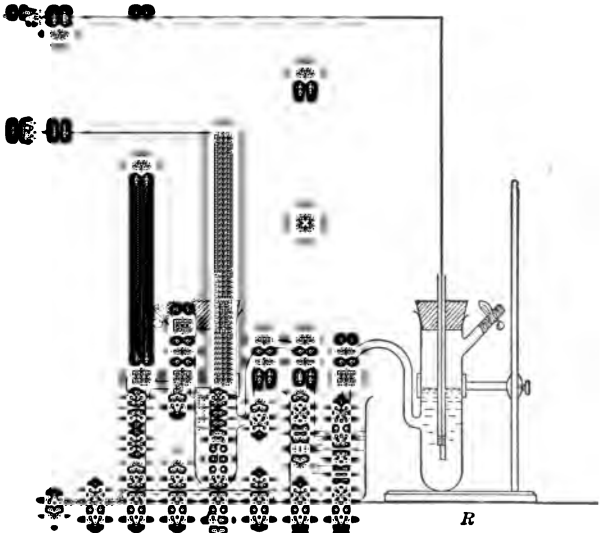
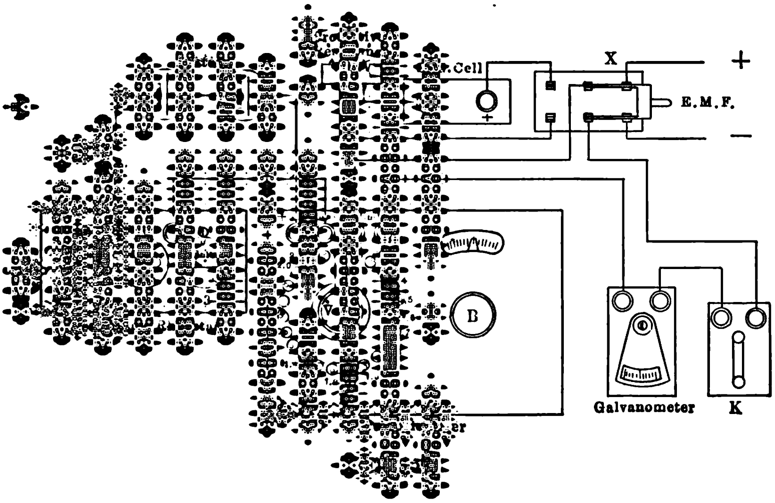
[1] In case a potentiometer is not available, the slide-wire arrangement described in Experiment 23 may be used.


R

[illegible]


Cell
X
+
E.M.F.
−
B
Galvanometer
K

[illegible]

Method of Procedure:

Clean and dry the half-cell elements, and the 50-c.c. beaker. Place about 40 c.c. of normal KNO_3 in the latter.

Hold the finger over the tip of the half-cell marked N/1, and pour sufficient normal $AgNO_3$ into the vessel to fill it to a point just below the upper side-arm. Place the silver electrode[1] in position, and close the side-arm, then remove the finger from the tip. Make sure that the element does not leak.

Fill half-cell marked N/10 with tenth-normal $AgNO_3$ in like manner.

Place the more concentrated solution in position *L*, and the more dilute, in *R* (Fig. 27). Make all electrical connections as per the diagram (Fig. 28).

Set dials *A* and *B* on the potentiometer to correspond to the potential of the standard cell, see page 158. Close switch *S*, and throw double-pole switch *X* to connect with the standard cell.

Close *K* for an instant, and observe the deflection of the galvanometer. By suitably adjusting the resistance *R*[2] and tapping *K*, the current in the potentiometer circuit may be adjusted until the galvanometer gives no deflection when *K* is closed.

The potentiometer is now in condition to read electromotive force directly from its scale.

Turn dial *B* until it reads zero, and throw switch *X* to make connection with the E.M.F. to be measured.

Tap *K*, and note the deflection of the galvanometer. Adjust dial *A* until its position corresponds to an E.M.F. one step lower than the potential of the concentration cell, then adjust dial *B* until a balance is secured.

---

[1] In order that the silver electrodes may possess a uniform potential, they should be kept "short-circuited" in a solution of N/10 $AgNO_3$ when not in use.

[2] In the adjustable resistance shown, dial 1 is for rough, 2 for approximate, and 3 for accurate, adjustment. Turn 2 and 3 counter-clockwise as far as they will go, and adjust 1 until the current in the potentiometer circuit is slightly greater than the proper strength for balance; then diminish the current by turning 2 in a clockwise direction, and finally adjust it accurately by means of dial 3.

Record the electromotive force of the concentration cell:

(Ag : N/1 $AgNO_3$: N/1 KNO_3 : N/10 $AgNO_3$: Ag)

Fill half-cell marked N/100 with hundredth-normal $AgNO_3$, place it in position *R*, transferring the tenth-normal solution to position *L*. Connect *X* with the standard cell, and check the value of the potentiometer current.

Return *X* to the other position, measure and record the potential of the concentration cell:

(Ag : N/10 $AgNO_3$: N/1 KNO_3 : N/100 $AgNO_3$: Ag)

Replace the hundredth-normal solution with a solution of unknown strength, in element marked N/*x*, check the potenttiometer current, and measure and record the E.M.F. of the cell:

(Ag : N/10 $AgNO_3$: N/1 KNO_3 : N/*x* $AgNO_3$: Ag)

Replace the solution of unknown strength with cell marked AgCl, containing a solution made up as follows:

Fill the cell with N/10 KCl, and add one or two drops of N/1 $AgNO_3$, in order to produce a saturated solution of AgCl in the cell. Place the N/100 $AgNO_3$ cell in position *L*.

Check the potentiometer, and measure and record the E.M.F. of the cell: (Ag : N/100 $AgNO_3$: N/1 KNO_3 : saturated AgCl in N/10 KCl : Ag).

Disconnect both wires from the standard cell, and open switches *S* and *X*.

Remove the solutions from the half-cell elements, pour them into the silver residue bottle, and rinse the cells with distilled water. Open all cocks, and invert the cells in their supports, so that they will drain.

Wash the electrodes, connect them all together by means of a piece of copper wire, and leave them in a tenth-normal $AgNO_3$ solution, contained in the 100-c.c. beaker.

Observations and Measurements:

A. E.M.F. of cell:
Ag : N $AgNO_3$: N KNO_3 : N/10 $AgNO_3$: Ag ......
B. E.M.F. of cell:
Ag : N/10 $AgNO_3$: N KNO_3: N/100 $AgNO_3$: Ag ......
C. E.M.F. of cell:
Ag : N/10 $AgNO_3$: N KNO_3 : N/x $AgNO_3$: Ag ......
D. E.M.F. of cell:
Ag : N/100 $AgNO_3$: N KNO_3 : saturated AgCl
in N/10 KNO_3 : Ag ......
Room temperature, ...

Calculations:

1. By means of the equation for electromotive force, as applied to concentration cells, calculate what the potentials should be in cases A and B, and compare the calculated with the observed values. See Table 11, Appendix.

2. Substituting the observed potential, case C, in the same equation, calculate the concentration of the unknown $AgNO_3$ solution. Express in terms of normality.

3. In like manner, calculate the concentration of AgCl in case D. This is the solubility of AgCl.

TABLES

TABLE 1

VAPOR PRESSURE OF WATER IN MM. MERCURY

At Low Temperatures

4°	6.1	19°	16.5
5	6.5	20	17.5
6	7.0	21	18.7
7	7.5	22	19.8
8	8.0	23	21.1
9	8.6	24	22.4
10	9.2	25	23.8
11	9.8	26	25.2
12	10.5	27	26.7
13	11.2	28	28.4
14	12.0	29	30.1
15	12.8	30	31.8
16	13.6	31	33.7
17	14.5	32	35.7
18	15.5	33	37.7

TABLE 2

Vapor Pressure of Water in mm. Mercury

At High Temperatures

Boiling Point	Pressure	Boiling Point	Pressure
90	526.0	97	682.1
	20.3		25.2
91	546.3	98	707.3
	20.9		26.0
92	567.2	99	733.3
	21.6		26.7
93	588.8	100	760.0
	22.2		27.6
94	611.0	101	787.6
	23.0		28.4
95	634.0	102	816.0
	23.7		29.3
96	657.7	103	845.3
	24.4		30.1
97	682.1	104	875.4

TABLE 3

Refractive Index of Water for Sodium Light at Different Temperatures

Temperature ° C.	Refractive Index	Temperature ° C.	Refractive Index
0	1.33395	22	1.33283
5	1.33388	23	1.33274
10	1.33368	24	1.33264
15	1.33337	25	1.33254
16	1.33330	26	1.33243
17	1.33323	27	1.33231
18	1.33316	28	1.33219
19	1.33308	29	1.33206
20	1.33300	30	1.33192
21	1.33292		

TABLE 4

DENSITIES (d_4) OF VARIOUS LIQUIDS

A. MERCURY (Regnault-Broch)

0°	13.5956	8	13.5759	15	13.5586	40	13.4974
1	13.5931	9	13.5734	16	13.5562	50	13.4731
2	13.5907	10	13.5709	17	13.5537	60	13.4488
3	13.5882	11	13.5685	18	13.5513	70	13.4246
4	13.5857	12	13.5660	19	13.5488	80	13.4005
5	13.5833	13	13.5635	20	13.5463	90	13.3764
6	13.5808	14	13.5611	30	13.5218	100	13.3524
7	13.5783						

B. WATER (Thiesen, Scheel, Marek)

0°	0.9999	5	1.0000	10	0.9997	60	0.9833
1	0.9999	6	1.0000	20	0.9982	70	0.9779
2	1.0000	7	0.9999	30	0.9957	80	0.9719
3	1.0000	8	0.9999	40	0.9923	90	0.9655
4	1.0000*	9	0.9998	50	0.9881	B.P.100	0.9586

C. NITROBENZENE (van Klooster)

0°	1.224	30	1.194	60	1.164	90	1.133
10	1.214	40	1.184	70	1.154	100	1.123
20	1.204	50	1.174	80	1.143		

D. CHLOROBENZENE (Jaeger)

0°	1.128	30	1.095	60	1.062	90	1.029
10	1.117	40	1.084	70	1.051	100	1.018
20	1.106	50	1.073	80	1.040		

E. Bromobenzene (Jaeger)

0°	1.520	30	1.480^5	60	1.440	90	1.400
10	1.507	40	1.467	70	1.427	100	1.387
20	1.494	50	1.453^5	80	1.413^5		

F. Carbon tetrachloride (Jaeger)

0°	1.632	30	1.576	50	1.536	70	1.494
10	1.615	40	1.555	60	1.516	B.P. 76	1.480
20	1.597						

TABLE 5

Freezing-point Constants (per 100 Grams of Solvent)

Substance	Freezing Point at 760 mm.	Freezing-point Constant K_F
Water	0°	18.6°
Acetic acid	17	39
Benzene	5.5	51.2
Naphthalene	80.1	69
Phenol	38	70

TABLE 6

Boiling-point Constants (per 100 Grams of Solvent) [1]

Substance	Boiling Point at 760 mm.	Boiling-point Constant K_B
Water	100°	5.2°
Acetic acid	118.5	32.8
Benzene	80.15	25.8
Carbon tetrachloride	76.5	50.5
Chloroform	60.2	36.4
Ether	34.4	22.1
Ethyl acetate	77.1	29.0

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[1] From Rosanoff and Dunphy, J. Am. Chem. Soc., *36*, 1415 (1914).

TABLE 7

Density, Percentage (Grams of HCl per 100 Grams of Solution) and Normality of Aqueous Solutions of Hydrochloric Acid

d_4^{15}	% HCl	Normality	d_4^{15}	% HCl	Normality
1.000	0.16	0.044	1.100	20.01	6.035
1.005	1.15	0.317	1.105	20.97	6.354
1.010	2.14	0.593	1.110	21.92	6.671
1.015	3.12	0.868	1.115	22.86	6.989
1.020	4.13	1.155	1.120	23.82	7.315
1.025	5.15	1.447	1.125	24.78	7.644
1.030	6.15	1.737	1.130	25.75	7.978
1.035	7.15	2.029	1.135	26.70	8.309
1.040	8.16	2.327	1.140	27.66	8.646
1.045	9.16	2.625	1.145	28.61	8.982
1.050	10.17	2.928	1.150	29.57	9.324
1.055	11.18	3.234	1.155	30.55	9.675
1.060	12.19	3.543	1.160	31.52	10.03
1.065	13.19	3.852	1.165	32.49	10.38
1.070	14.17	4.157	1.170	33.46	10.73
1.075	15.16	4.470	1.175	34.42	11.09
1.080	16.15	4.782	1.180	35.39	11.45
1.085	17.13	5.096	1.185	36.31	11.80
1.090	18.11	5.413	1.190	37.23	12.15
1.095	19.06	5.723	1.195	38.16	12.50
1.100	20.01	6.035	1.200	39.11	12.87

TABLE 8

Density and Specific Heat of Different Substances at Room Temperature

Substance	Density	Specific Heat
Glass	2.5	0.19
Mercury	13.5	0.034
Brass	8.5	0.094
Copper	8.8	0.092
Gold	19.3	0.0315
Iron	7.85	0.115
Nickel	9.1	0.109
Platinum	20.3	0.032
Steel	....	0.116

TABLE 9

SPECIFIC CONDUCTANCE OF FIFTIETH-NORMAL KCl

(containing 1.492 Grams per Liter) at
Different Temperatures

Temperature	Conductance	Temperature	Conductance
10°	0.001521	24°	0.002712
15	0.002243	25	0.002765
18	0.002397	26	0.002819
20	0.002501	30	0.003036

TABLE 10

EQUIVALENT CONDUCTANCES AT INFINITE DILUTION OF WEAK ACIDS at 25°

Acetic acid	389	Oxalic acid	422.7
Benzoic acid	381	Succinic acid	381
Cinnamic acid	378.5		

TABLE 11

TRANSPORT NUMBERS OF CATIONS AT 18°

	N/100	N/10	N/1
$AgNO_3$	.471	.471	.465
KNO_3	——	.502	——
KCl	.496	.495	——
HNO_3	——	.855	——
HCl	.833	.835	——
$CuSO_4$	.375	.373	.330
H_2SO_4	.824	.824	——

MAXIMUM IONIC CONDUCTANCES AT 18°

Na^+	43.5	Cu^{++}	46.0
Ag^+	54.3	Cl^-	65.5
K^+	64.6	NO_3^-	61.7
H^+	315.0	SO_4^{--}	68.0

TABLE 12

REDUCTION OF BAROMETER READINGS TO 0°

When the height of the mercury column has been measured with a glass or a brass scale, the length of which is correct at 0°, the mercury and the scale being at $t°$, the observed height is reduced to 0° by *subtracting* the value given in the table corresponding to the temperature and height.

	Glass Scale (Bunsen)						Brass Scale (Delcros)				
Temperature.	Barometer Reading in mm.					Temperature.	Barometer Reading in mm.				
	740	750	760	770	780		740	750	760	770	780
1°	0.13	0.13	0.13	0.13	0.14	1°	0.12	0.12	0.12	0.13	0.13
2	0.26	0.26	0.26	0.27	0.27	2	0.24	0.25	0.25	0.25	0.25
3	0.38	0.39	0.39	0.40	0.41	3	0.36	0.37	0.37	0.38	0.38
4	0.51	0.52	0.53	0.53	0.54	4	0.48	0.49	0.50	0.50	0.51
5	0.64	0.65	0.66	0.67	0.68	5	0.60	0.61	0.62	0.63	0.64
6	0.77	0.78	0.79	0.80	0.81	6	0.72	0.73	0.74	0.75	0.76
7	0.90	0.91	0.92	0.93	0.95	7	0.85	0.86	0.87	0.88	0.89
8	1.02	1.04	1.05	1.07	1.08	8	0.97	0.98	0.99	1.01	1.02
9	1.15	1.17	1.18	1.20	1.21	9	1.09	1.10	1.12	1.13	1.15
10	1.28	1.30	1.31	1.33	1.35	10	1.21	1.22	1.24	1.26	1.27
11	1.41	1.43	1.45	1.46	1.48	11	1.33	1.35	1.36	1.38	1.40
12	1.53	1.56	1.58	1.60	1.62	12	1.45	1.47	1.49	1.51	1.53
13	1.66	1.69	1.71	1.73	1.75	13	1.57	1.59	1.61	1.63	1.65
14	1.79	1.81	1.84	1.86	1.89	14	1.69	1.71	1.73	1.76	1.78
15	1.92	1.94	1.97	2.00	2.02	15	1.81	1.83	1.86	1.88	1.91
16	2.05	2.07	2.10	2.13	2.16	16	1.93	1.96	1.98	2.01	2.03
17	2.17	2.20	2.23	2.26	2.29	17	2.05	2.08	2.10	2.13	2.16
18	2.30	2.33	2.36	2.39	2.43	18	2.17	2.20	2.23	2.26	2.29
19	2.43	2.46	2.49	2.53	2.56	19	2.29	2.32	2.35	2.38	2.41
20	2.56	2.59	2.62	2.66	2.69	20	2.41	2.44	2.47	2.51	2.54
21	2.08	2.72	2.76	2.79	2.83	21	2.53	2.56	2.60	2.63	2.67
22	2.81	2.85	2.89	2.92	2.96	22	2.65	2.69	2.72	2.76	2.79
23	2.94	2.98	3.02	3.06	3.10	23	2.77	2.81	2.84	2.88	2.92
24	3.06	3.11	3.15	3.19	3.23	24	2.89	2.93	2.97	3.01	3.05
25	3.19	3.23	3.28	3.32	3.36	25	3.01	3.05	3.09	3.13	3.17
26	3.32	3.36	3.41	3.45	3.50	26	3.13	3.17	3.21	3.26	3.30
27	3.45	3.49	3.54	3.59	3.63	27	3.25	3.29	3.34	3.38	3.42
28	3.57	3.62	3.67	3.72	3.77	28	3.37	3.41	3.46	3.51	3.55
29	3.70	3.75	3.80	3.85	3.90	29	3.49	3.54	3.58	3.63	3.68
30	3.83	3.88	3.93	3.98	4.03	30	3.61	3.66	3.71	3.75	3.80

TABLE 13

FOUR PLACE LOGARITHMS

											Proportionality factors								
	0	1	2	3	4	5	6	7	8	9	1	2	3	4	5	6	7	8	9
10	0000	0043	0086	0128	0170	0212	0253	0294	0334	0374	4	8	12	17	21	25	29	33	37
11	0414	0453	0492	0531	0569	0607	0645	0682	0719	0755	4	8	11	15	19	23	26	30	34
12	0792	0828	0864	0899	0934	0969	1004	1038	1072	1106	3	7	10	14	17	21	24	28	31
13	1139	1173	1206	1239	1271	1303	1335	1367	1399	1430	3	6	10	13	16	19	23	26	29
14	1461	1492	1523	1553	1584	1614	1644	1673	1703	1732	3	6	9	12	15	18	21	24	27
15	1761	1790	1818	1847	1875	1903	1931	1959	1987	2014	3	6	8	11	14	17	20	22	25
16	2041	2068	2095	2122	2148	2175	2201	2227	2253	2279	3	5	8	11	13	16	18	21	24
17	2304	2330	2355	2380	2405	2430	2455	2480	2504	2529	2	5	7	10	12	15	17	20	22
18	2553	2577	2601	2625	2648	2672	2695	2718	2742	2765	2	5	7	9	12	14	16	19	21
19	2788	2810	2833	2856	2878	2900	2923	2945	2967	2989	2	4	7	9	11	13	16	18	20
20	3010	3032	3054	3075	3096	3118	3139	3160	3181	3201	2	4	6	8	11	13	15	17	19
21	3222	3243	3263	3284	3304	3324	3345	3365	3385	3404	2	4	6	8	10	12	14	16	18
22	3424	3444	3464	3483	3502	3522	3541	3560	3579	3598	2	4	6	8	10	12	14	15	17
23	3617	3636	3655	3674	3692	3711	3729	3747	3766	3784	2	4	6	7	9	11	13	15	17
24	3802	3820	3838	3856	3874	3892	3909	3927	3945	3962	2	4	5	7	9	11	12	14	16
25	3979	3997	4014	4031	4048	4065	4082	4099	4116	4133	2	3	5	7	9	10	12	14	15
26	4150	4166	4183	4200	4216	4232	4249	4265	4281	4298	2	3	5	7	8	10	11	13	15
27	4314	4330	4346	4362	4378	4393	4409	4425	4440	4456	2	3	5	6	8	9	11	13	14
28	4472	4487	4502	4518	4533	4548	4564	4579	4594	4609	2	3	5	6	8	9	11	12	14
29	4624	4639	4654	4669	4683	4698	4713	4728	4742	4757	1	3	4	6	7	9	10	12	13
30	4771	4786	4800	4814	4829	4843	4857	4871	4886	4900	1	3	4	6	7	9	10	11	13
31	4914	4928	4942	4955	4969	4983	4997	5011	5024	5038	1	3	4	6	7	8	10	11	12
32	5051	5065	5079	5092	5105	5119	5132	5145	5159	5172	1	3	4	5	7	8	9	11	12
33	5185	5198	5211	5224	5237	5250	5263	5276	5289	5302	1	3	4	5	6	8	9	10	12
34	5315	5328	5340	5353	5366	5378	5391	5403	5416	5428	1	3	4	5	6	8	9	10	11
35	5441	5453	5465	5478	5490	5502	5514	5527	5539	5551	1	2	4	5	6	7	9	10	11
36	5563	5575	5587	5599	5611	5623	5635	5647	5658	5670	1	2	4	5	6	7	8	10	11
37	5682	5694	5705	5717	5729	5740	5752	5763	5775	5786	1	2	3	5	6	7	8	9	10
38	5798	5809	5821	5832	5843	5855	5866	5877	5888	5899	1	2	3	5	6	7	8	9	10
39	5911	5922	5933	5944	5955	5966	5977	5988	5999	6010	1	2	3	4	5	7	8	9	10
40	6021	6031	6042	6053	6064	6075	6085	6096	6107	6117	1	2	3	4	5	6	8	9	10
41	6128	6138	6149	6160	6170	6180	6191	6201	6212	6222	1	2	3	4	5	6	7	8	9
42	6232	6243	6253	6263	6274	6284	6294	6304	6314	6325	1	2	3	4	5	6	7	8	9
43	6335	6345	6355	6365	6375	6385	6395	6405	6415	6425	1	2	3	4	5	6	7	8	9
44	6435	6444	6454	6464	6474	6484	6493	6503	6513	6522	1	2	3	4	5	6	7	8	9
45	6532	6542	6551	6561	6571	6580	6590	6599	6609	6618	1	2	3	4	5	6	7	8	9
46	6628	6637	6646	6656	6665	6675	6684	6693	6702	6712	1	2	3	4	5	6	7	7	8
47	6721	6730	6739	6749	6758	6767	6776	6785	6794	6803	1	2	3	4	5	5	6	7	8
48	6812	6821	6830	6839	6848	6857	6866	6875	6884	6893	1	2	3	4	4	5	6	7	8
49	6902	6911	6920	6928	6937	6946	6955	6964	6972	6981	1	2	3	4	4	5	6	7	8
50	6990	6998	7007	7016	7024	7033	7042	7050	7059	7067	1	2	3	3	4	5	6	7	8
51	7076	7084	7093	7101	7110	7118	7126	7135	7143	7152	1	2	3	3	4	5	6	7	8
52	7160	7168	7177	7185	7193	7202	7210	7218	7226	7235	1	2	2	3	4	5	6	7	7
53	7243	7251	7259	7267	7275	7284	7292	7300	7308	7316	1	2	2	3	4	5	6	6	7
54	7324	7332	7340	7348	7356	7364	7372	7380	7388	7396	1	2	2	3	4	5	6	6	7

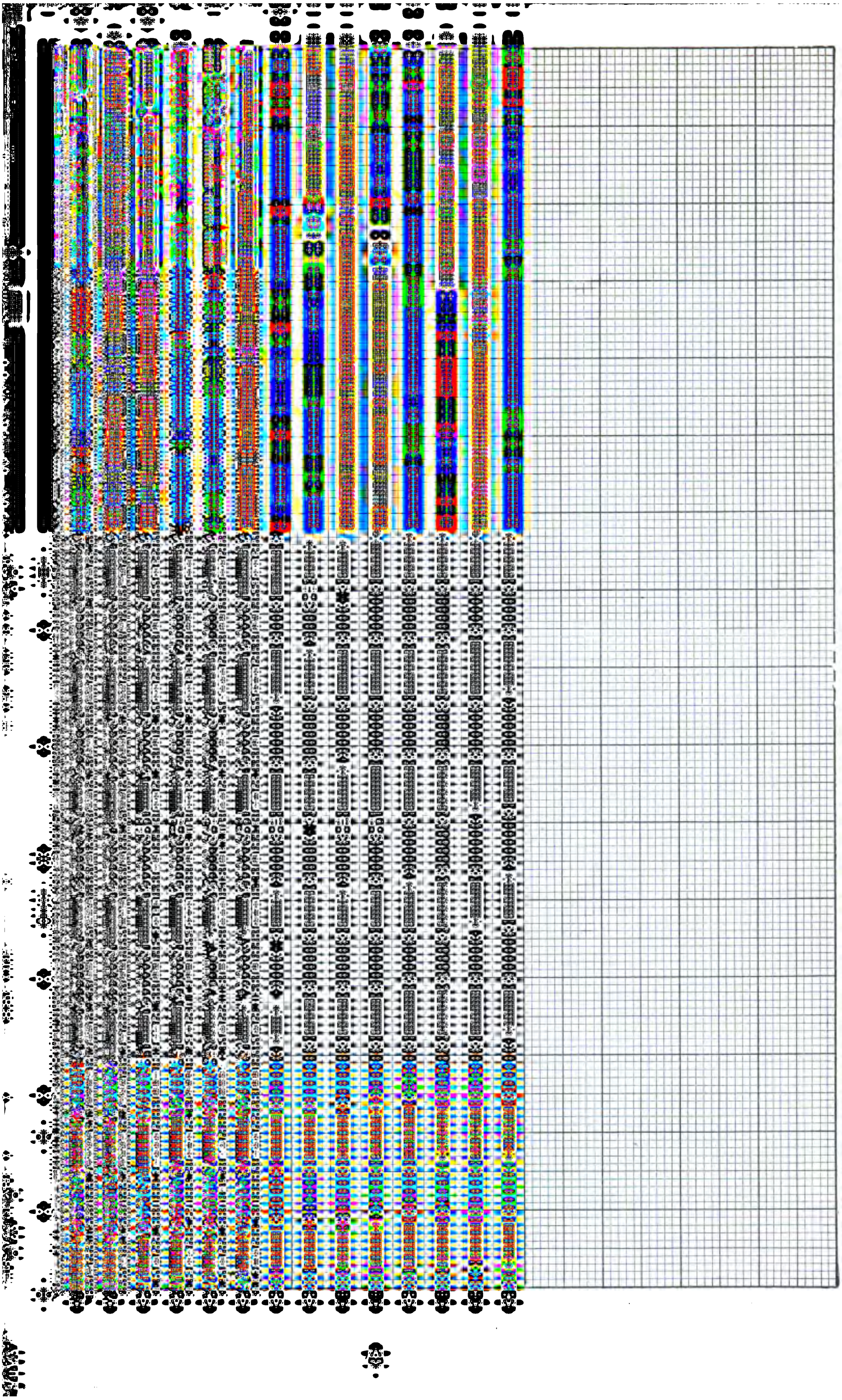
TABLE 13.—FOUR PLACE LOGARITHMS (*Continued*)

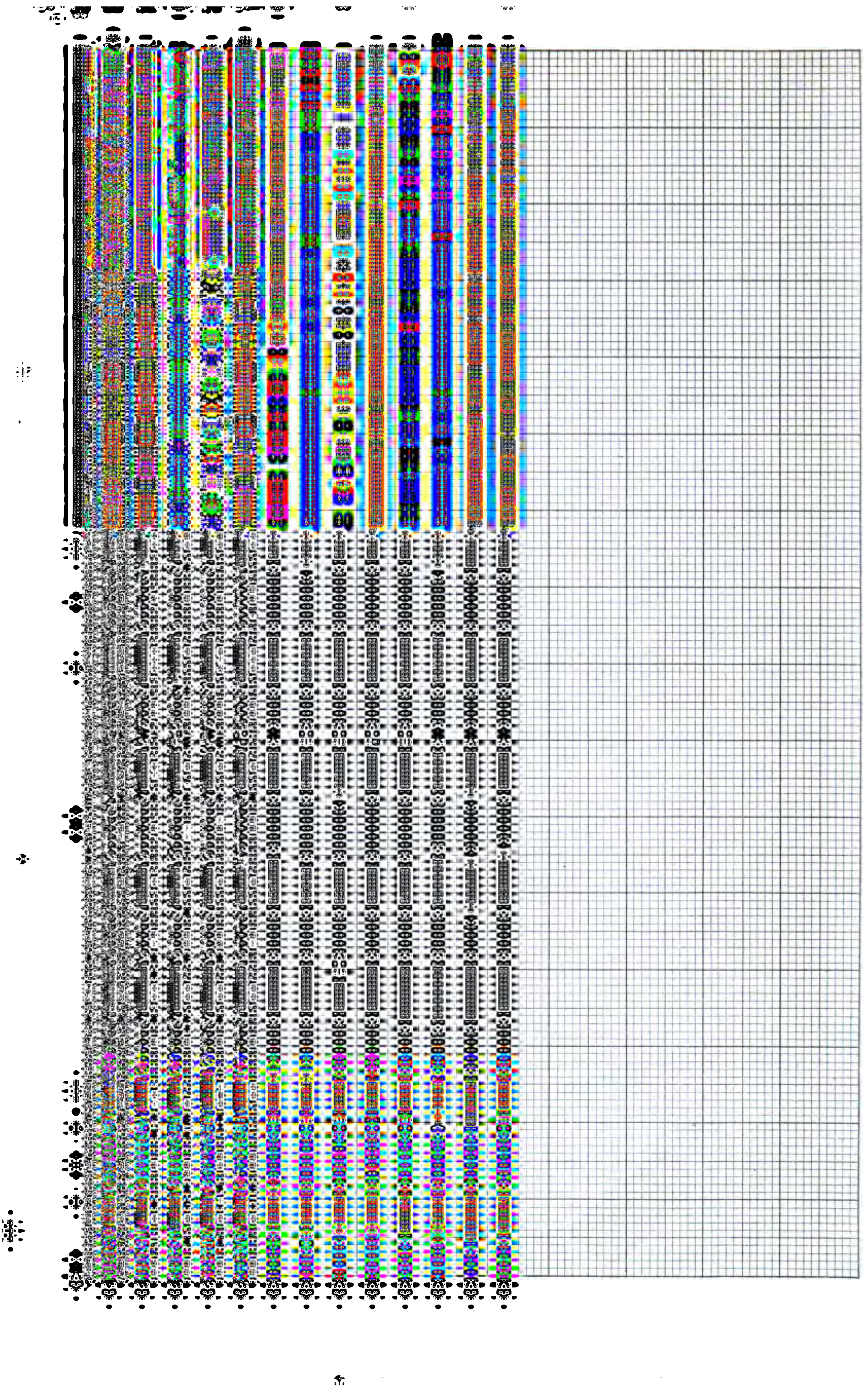
											Proportionality factors								
	0	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**
55	7404	7412	7419	7427	7435	7443	7451	7459	7466	7474	1	2	2	3	4	5	5	6	7
56	7482	7490	7497	7505	7513	7520	7528	7536	7543	7551	1	2	2	3	4	5	5	6	7
57	7559	7566	7574	7582	7589	7597	7604	7612	7619	7627	1	2	2	3	4	5	5	6	7
58	7634	7642	7649	7657	7664	7672	7679	7686	7694	7701	1	1	2	3	4	4	5	6	7
59	7709	7716	7723	7731	7738	7745	7752	7760	7767	7774	1	1	2	3	4	4	5	6	7
60	7782	7789	7796	7803	7810	7818	7825	7832	7839	7846	1	1	2	3	4	4	5	6	6
61	7853	7860	7868	7875	7882	7889	7896	7903	7910	7917	1	1	2	3	4	4	5	6	6
62	7924	7931	7938	7945	7952	7959	7966	7973	7980	7987	1	1	2	3	3	4	5	6	6
63	7993	8000	8007	8014	8021	8028	8035	8041	8048	8055	1	1	2	3	3	4	5	5	6
64	8062	8069	8075	8082	8089	8096	8102	8109	8116	8122	1	1	2	3	3	4	5	5	6
65	8129	8136	8142	8149	8156	8162	8169	8176	8182	8189	1	1	2	3	3	4	5	5	6
66	8195	8202	8209	8215	8222	8228	8235	8241	8248	8254	1	1	2	3	3	4	5	5	6
67	8261	8267	8274	8280	8287	8293	8299	8306	8312	8319	1	1	2	3	3	4	5	5	6
68	8325	8331	8338	8344	8351	8357	8363	8370	8376	8382	1	1	2	3	3	4	4	5	6
69	8388	8395	8401	8407	8414	8420	8426	8432	8439	8445	1	1	2	2	3	4	4	5	6
70	8451	8457	8463	8470	8476	8482	8488	8494	8500	8506	1	1	2	2	3	4	4	5	6
71	8513	8519	8525	8531	8537	8543	8549	8555	8561	8567	1	1	2	2	3	4	4	5	5
72	8573	8579	8585	8591	8597	8603	8609	8615	8621	8627	1	1	2	2	3	4	4	5	5
73	8633	8639	8645	8651	8657	8663	8669	8675	8681	8686	1	1	2	2	3	4	4	5	5
74	8692	8698	8704	8710	8716	8722	8727	8733	8739	8745	1	1	2	2	3	4	4	5	5
75	8751	8756	8762	8768	8774	8779	8785	8791	8797	8802	1	1	2	2	3	3	4	5	5
76	8808	8814	8820	8825	8831	8837	8842	8848	8854	8859	1	1	2	2	3	3	4	5	5
77	8865	8871	8876	8882	8887	8893	8899	8904	8910	8915	1	1	2	2	3	3	4	4	5
78	8921	8927	8932	8938	8943	8949	8954	8960	8965	8971	1	1	2	2	3	3	4	4	5
79	8976	8982	8987	8993	8998	9004	9009	9015	9029	9025	1	1	2	2	3	3	4	4	5
80	9031	9036	9042	9047	9053	9058	9063	9069	9074	9079	1	1	2	2	3	3	4	4	5
81	9085	9090	9096	9101	9106	9112	9117	9122	9128	9133	1	1	2	2	3	3	4	4	5
82	9138	9143	9149	9154	9159	9165	9170	9175	9180	9186	1	1	2	2	3	3	4	4	5
83	9191	9196	9201	9206	9212	9217	9222	9227	9232	9238	1	1	2	2	3	3	4	4	5
84	9243	9248	9253	9258	9263	9269	9274	9279	9284	9289	1	1	2	2	3	3	4	4	5
85	9294	9299	9304	9309	9315	9320	9325	9330	9335	9340	1	1	2	2	3	3	4	4	5
86	9345	9350	9355	9360	9365	9370	9375	9380	9385	9390	1	1	2	2	3	3	4	4	5
87	9395	9400	9405	9410	9415	9420	9425	9430	9435	9440	0	1	1	2	2	3	3	4	4
88	9445	9450	9455	9460	9465	9469	9474	9479	9484	9489	0	1	1	2	2	3	3	4	4
89	9494	9499	9504	9509	9513	9518	9523	9528	9533	9538	0	1	1	2	2	3	3	4	4
90	9542	9547	9552	9557	9562	9566	9571	9576	9581	9586	0	1	1	2	2	3	3	4	4
91	9590	9595	9600	9605	9609	9614	9619	9624	9628	9633	0	1	1	2	2	3	3	4	4
92	9638	9643	9647	9652	9657	9661	9666	9671	9675	9680	0	1	1	2	2	3	3	4	4
93	9685	9689	9694	9699	9703	9708	9713	9717	9722	9727	0	1	1	2	2	3	3	4	4
94	9731	9736	9741	9745	9750	9754	9759	9763	9768	9773	0	1	1	2	2	3	3	4	4
95	9777	9782	9786	9791	9795	9800	9805	9809	9814	9818	0	1	1	2	2	3	3	4	4
96	9823	9827	9832	9836	9841	9845	9850	9854	9859	9863	0	1	1	2	2	3	3	4	4
97	9868	9872	9877	9881	9886	9890	9894	9899	9903	9908	0	1	1	2	2	3	3	4	4
98	9912	9917	9921	9926	9930	9934	9939	9943	9948	9952	0	1	1	2	2	3	3	4	4
99	9956	9961	9965	9969	9974	9978	9983	9987	9991	9996	0	1	1	2	2	3	3	3	4

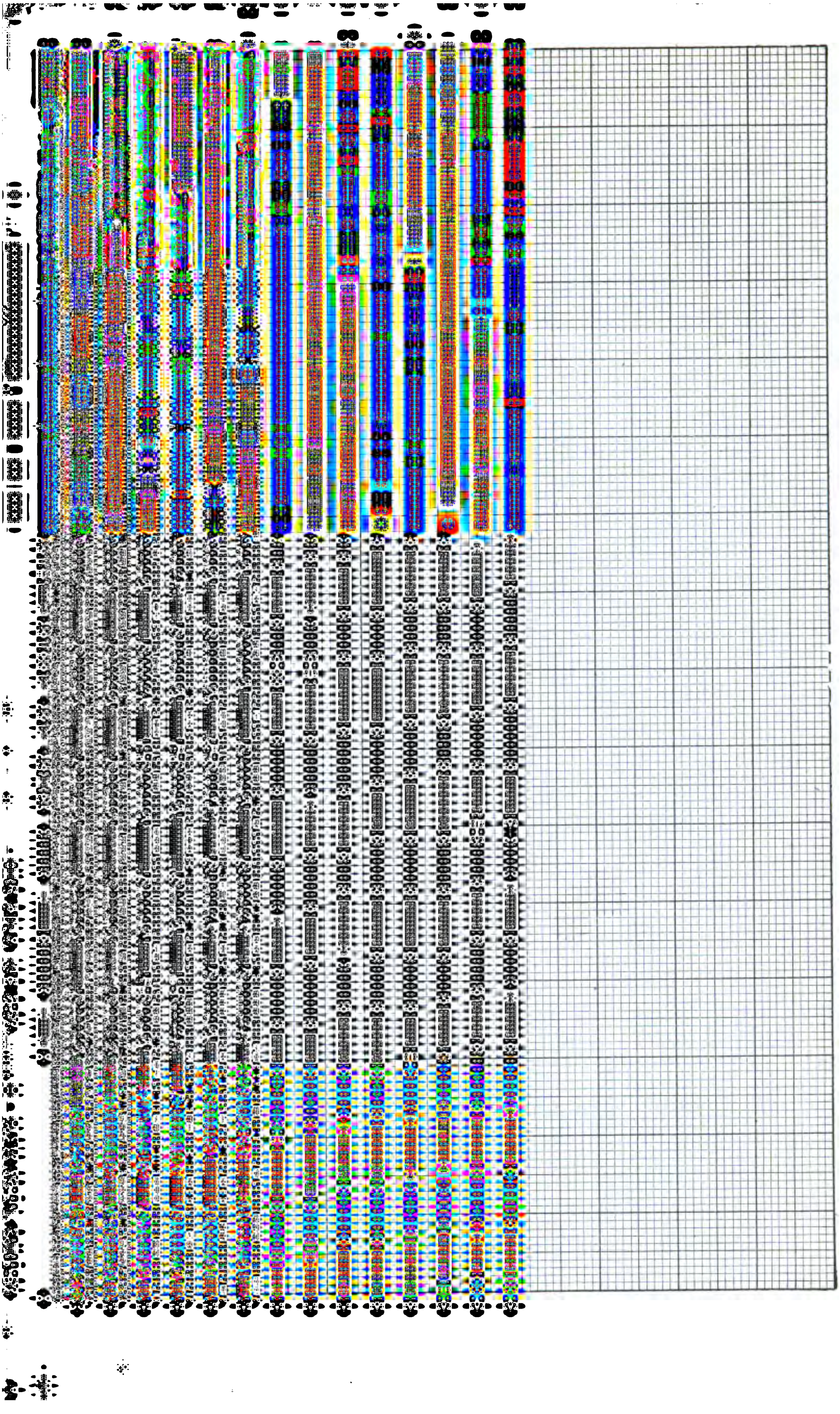
TABLE 14

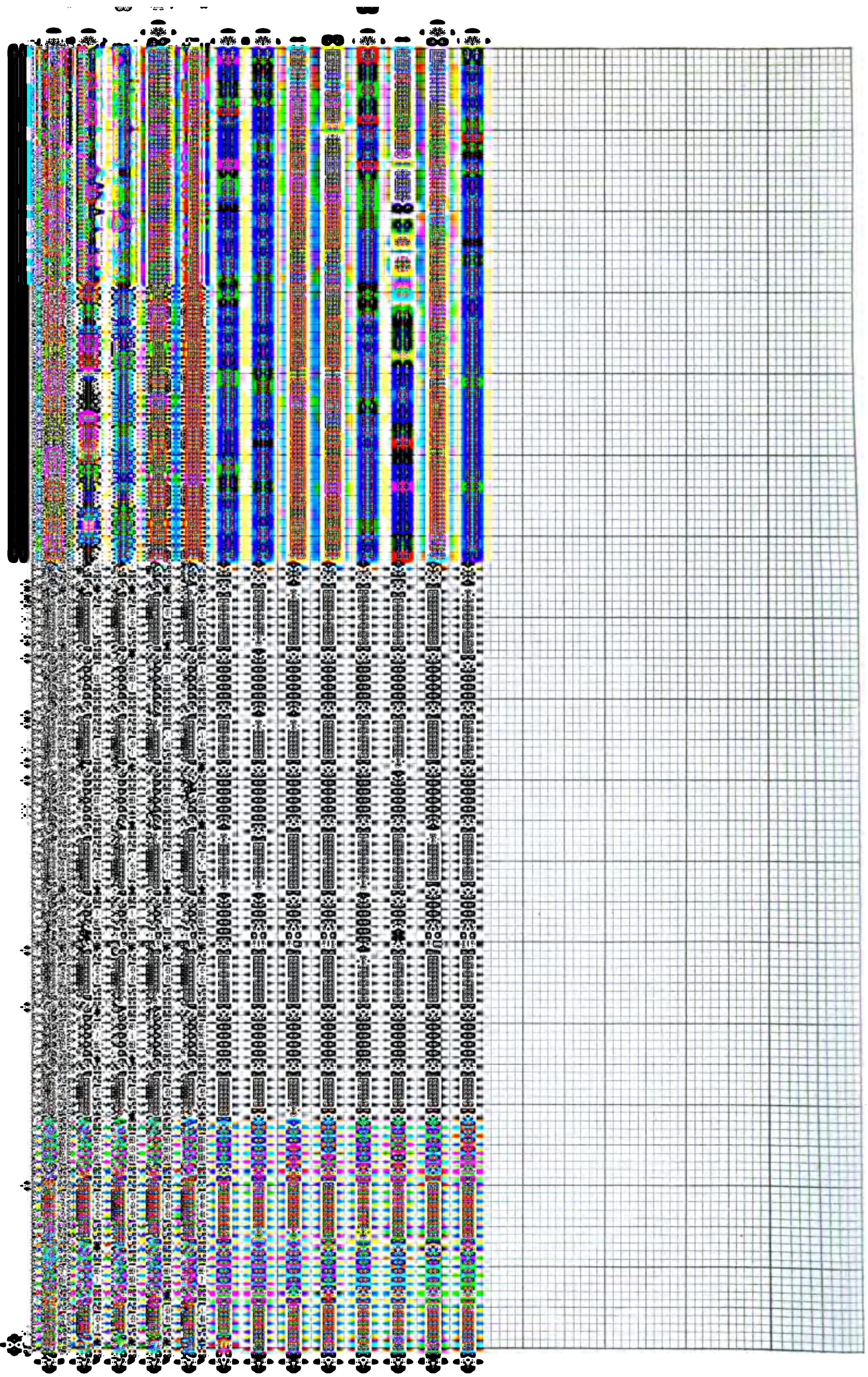
Atomic Weights of the More Common Elements

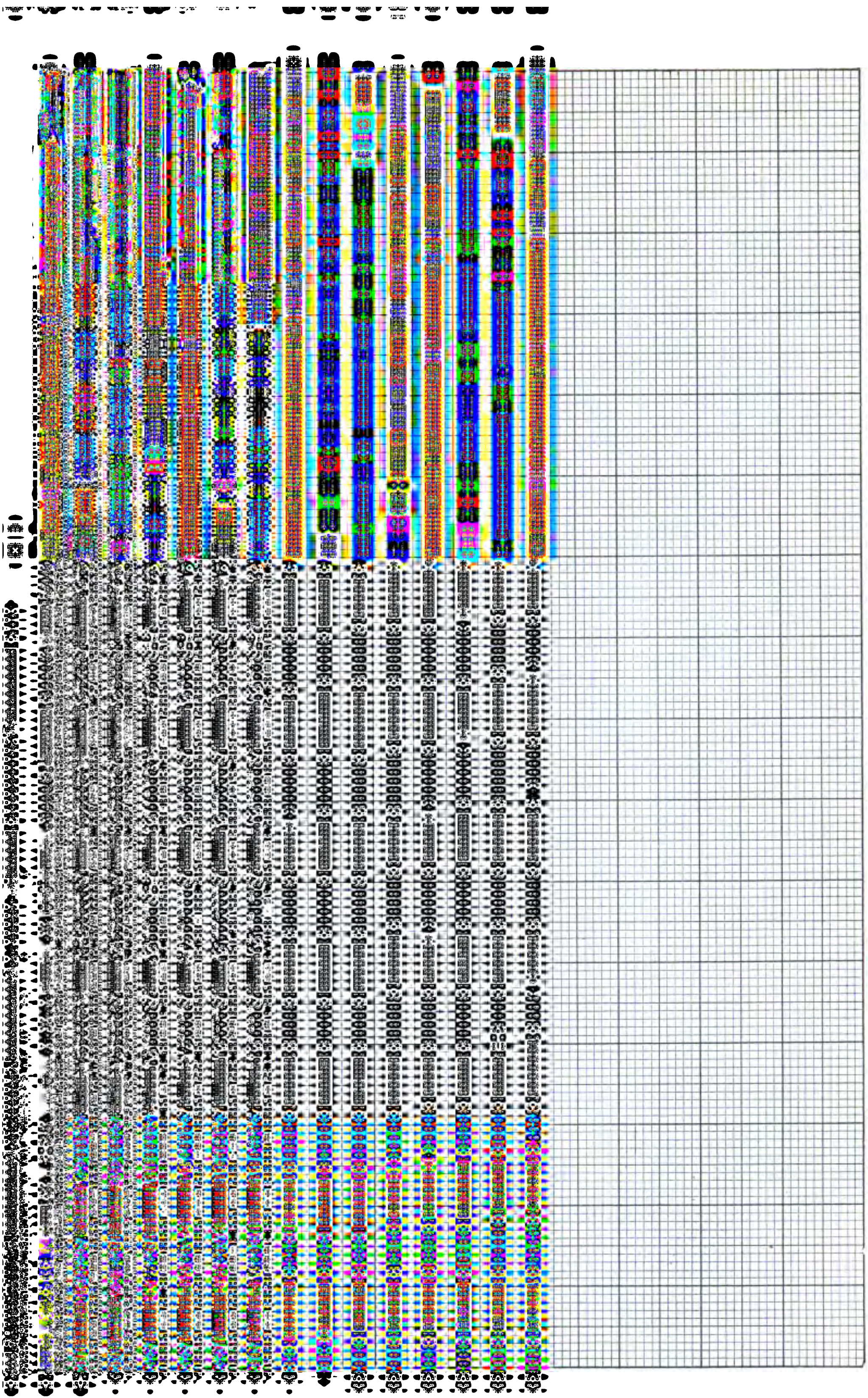
	Symbol	Atomic Weight		Symbol	Atomic Weight
Aluminium	Al	27.0	Manganese	Mn	54.9
Antimony	Sb	120.2	Mercury	Hg	200.6
Arsenic	As	75.0	Nickel	Ni	58.7
Barium	Ba	137.4	Nitrogen	N	14.0
Bismuth	Bi	209.0	Oxygen	O	16.0
Boron	B	10.9	Phosphorus	P	31.0
Bromine	Br	79.9	Platinum	Pt	195.2
Cadmium	Cd	112.4	Potassium	K	39.1
Calcium	Ca	40.1	Radium	Ra	226.0
Carbon	C	12.0	Selenium	Se	79.2
Chlorine	Cl	35.5	Silicon	Si	28.1
Chromium	Cr	52.0	Silver	Ag	107.9
Cobalt	Co	59.0	Sodium	Na	23.0
Copper	Cu	63.6	Strontium	Sr	87.6
Fluorine	F	19.0	Sulphur	S	32.1
Gold	Au	197.2	Tellurium	Te	127.5
Helium	He	4.0	Tin	Sn	118.7
Hydrogen	H	1.0	Titanium	Ti	48.1
Iodine	I	126.9	Tungsten	W	184.0
Iron	Fe	55.8	Uranium	U	238.2
Lead	Pb	207.2	Vanadium	V	51.0
Lithium	Li	6.9	Zinc	Zn	65.4
Magnesium	Mg	24.3	Zirconium	Zr	90.6

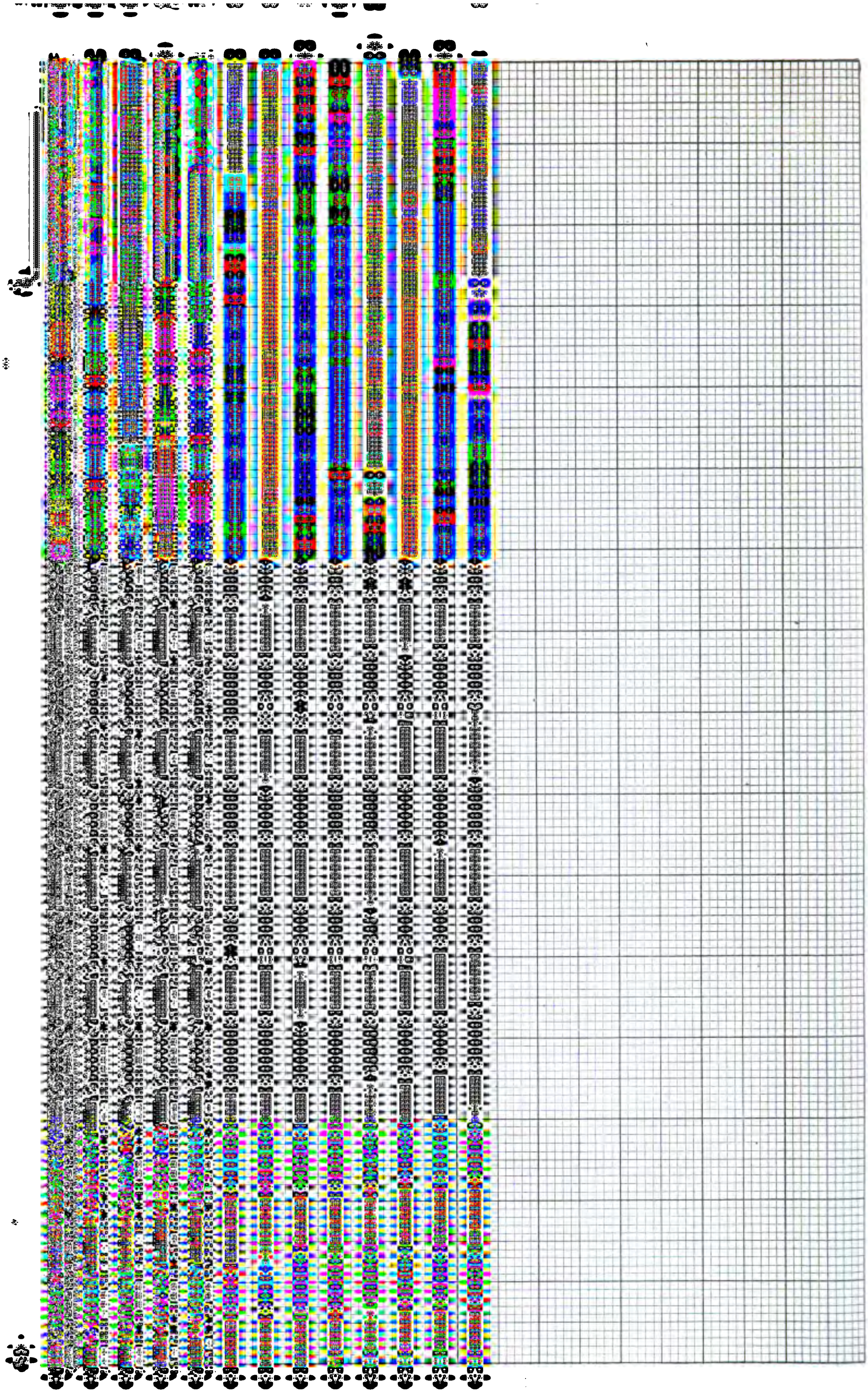


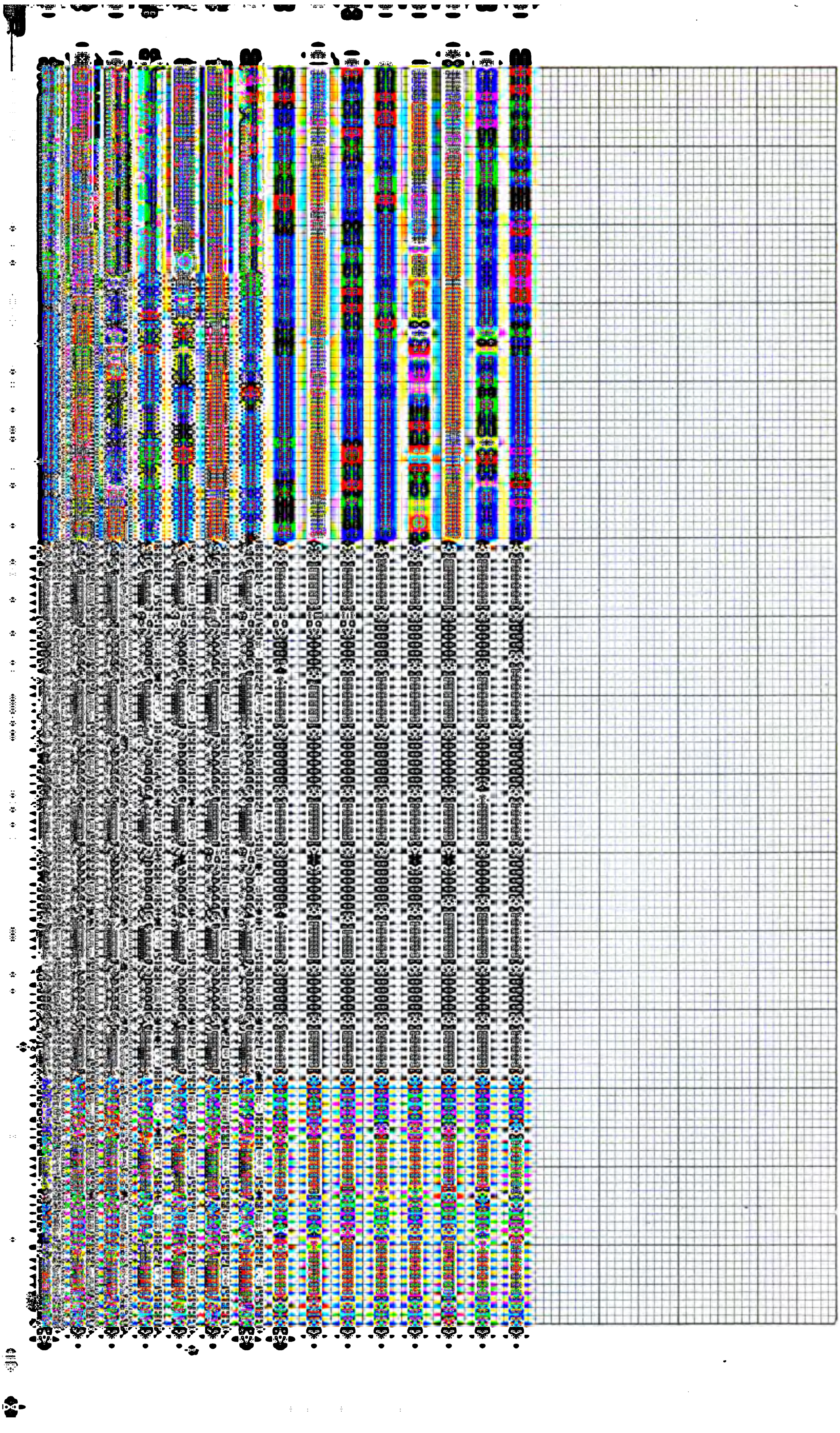


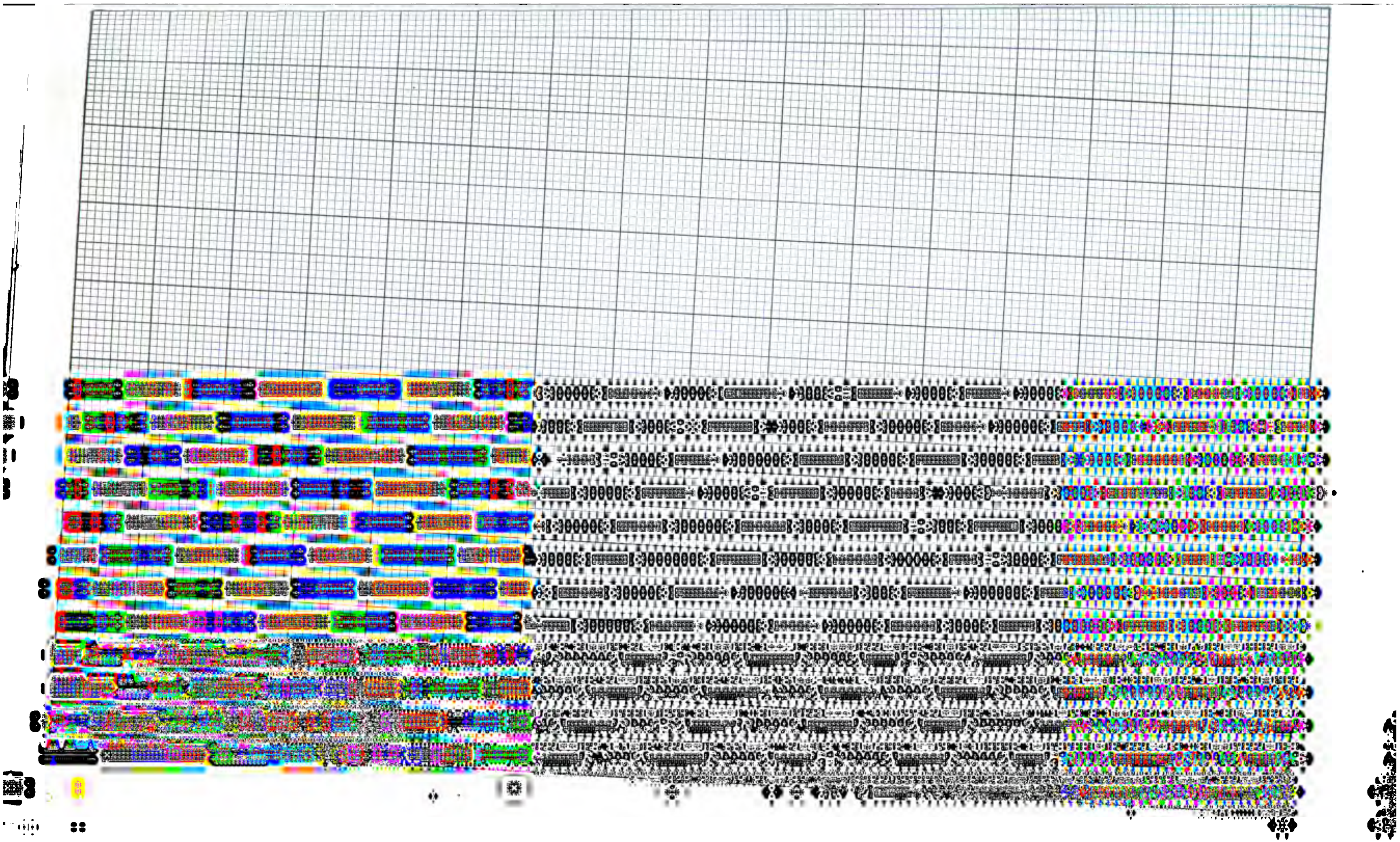


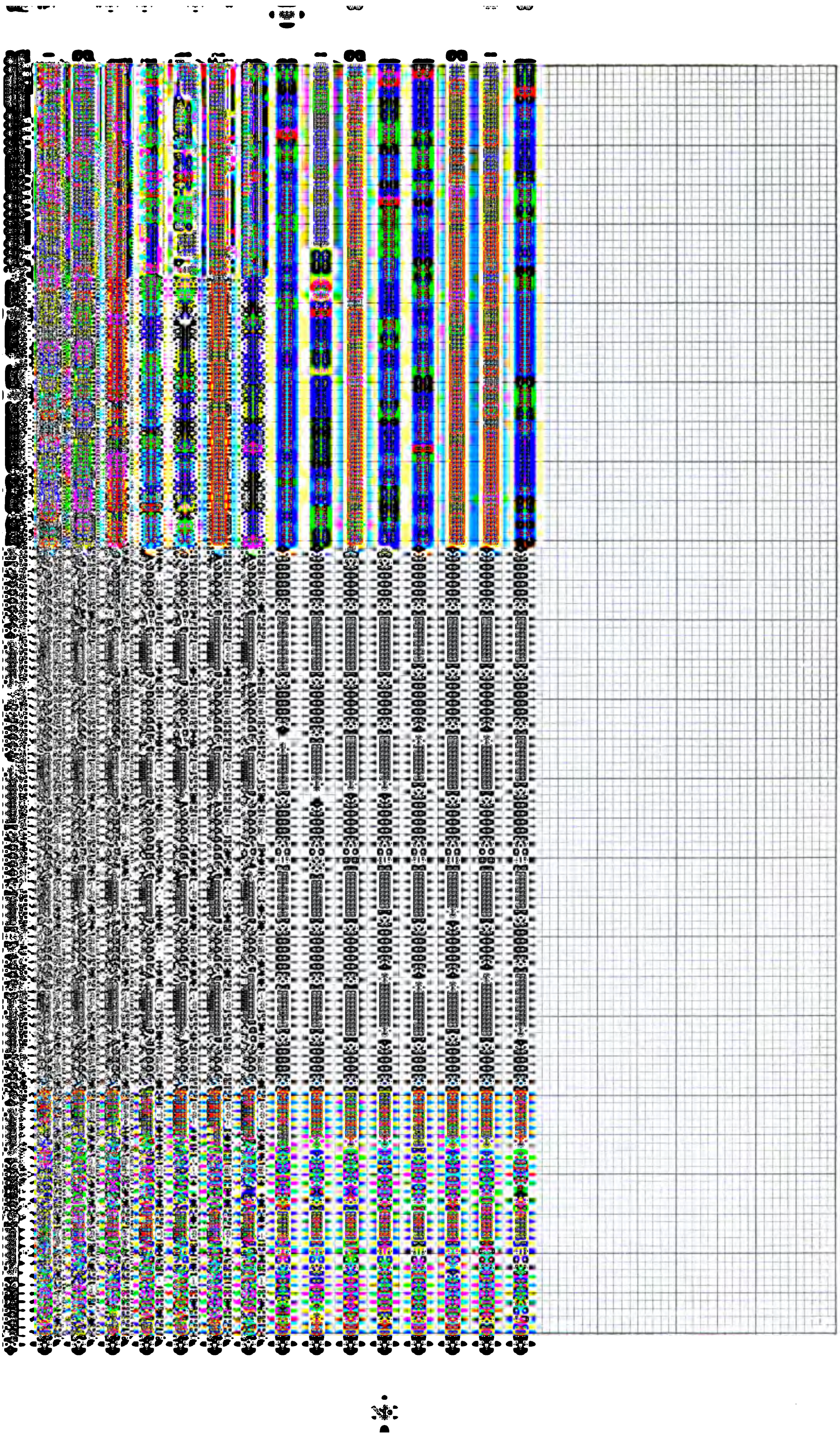


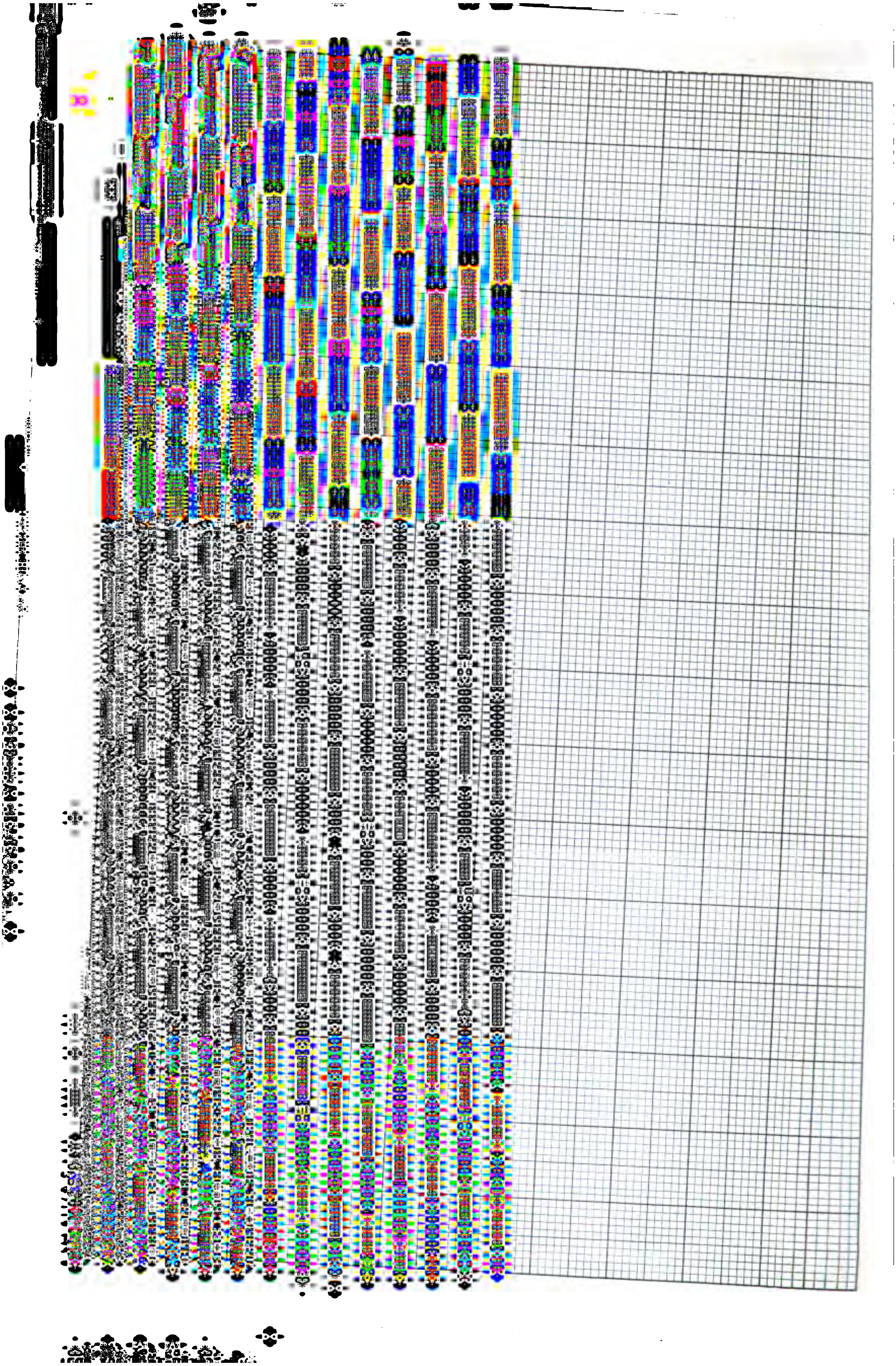


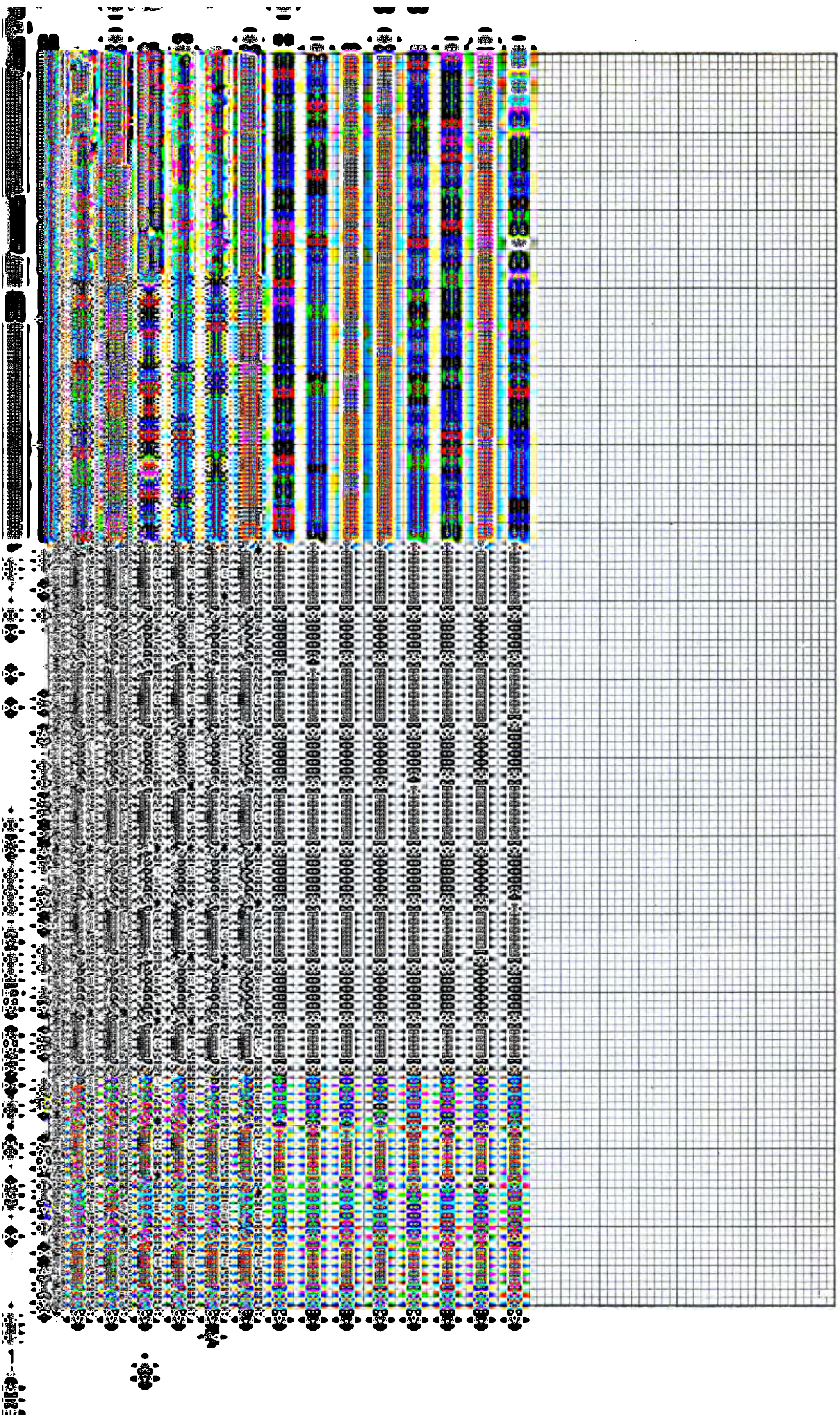


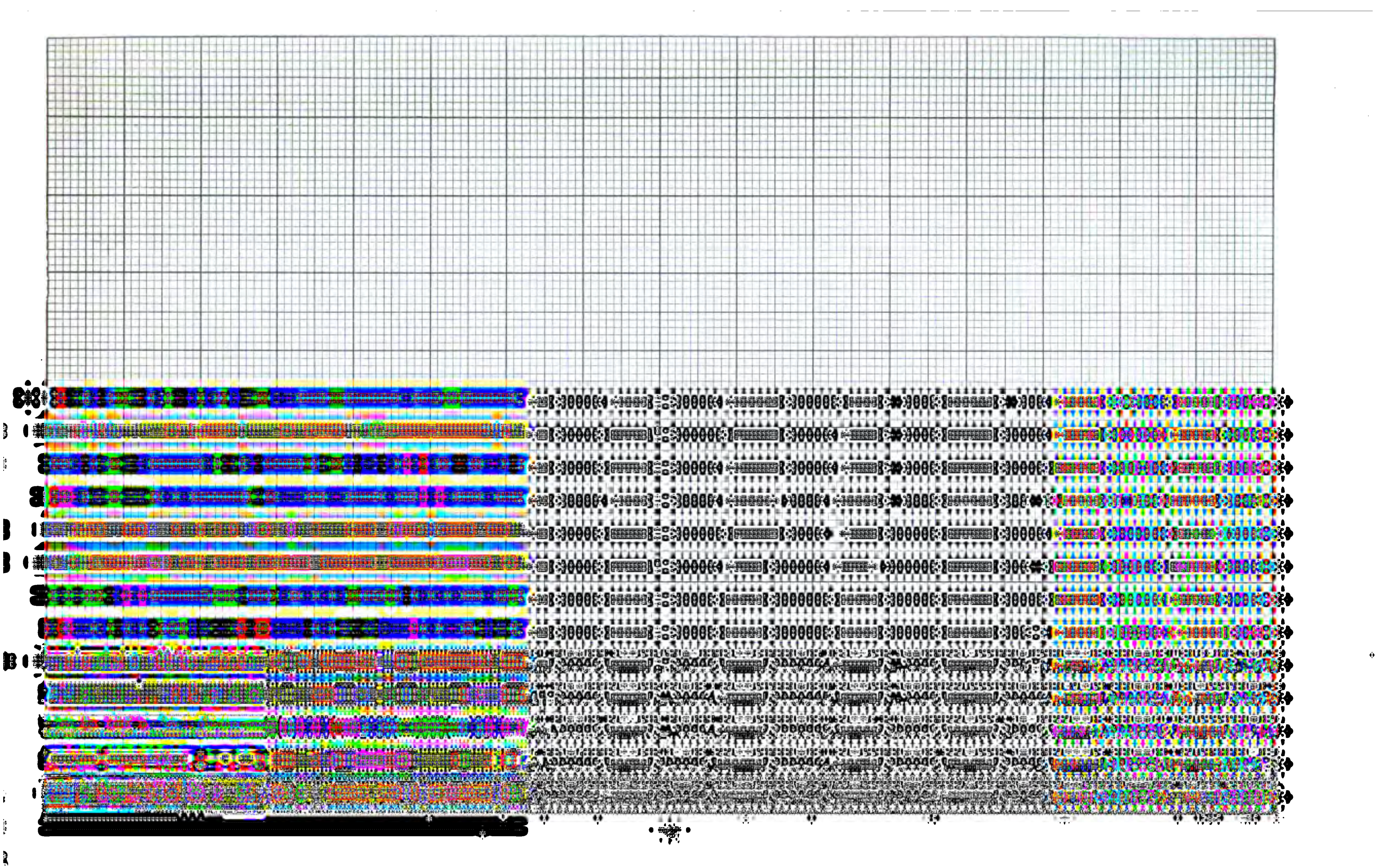


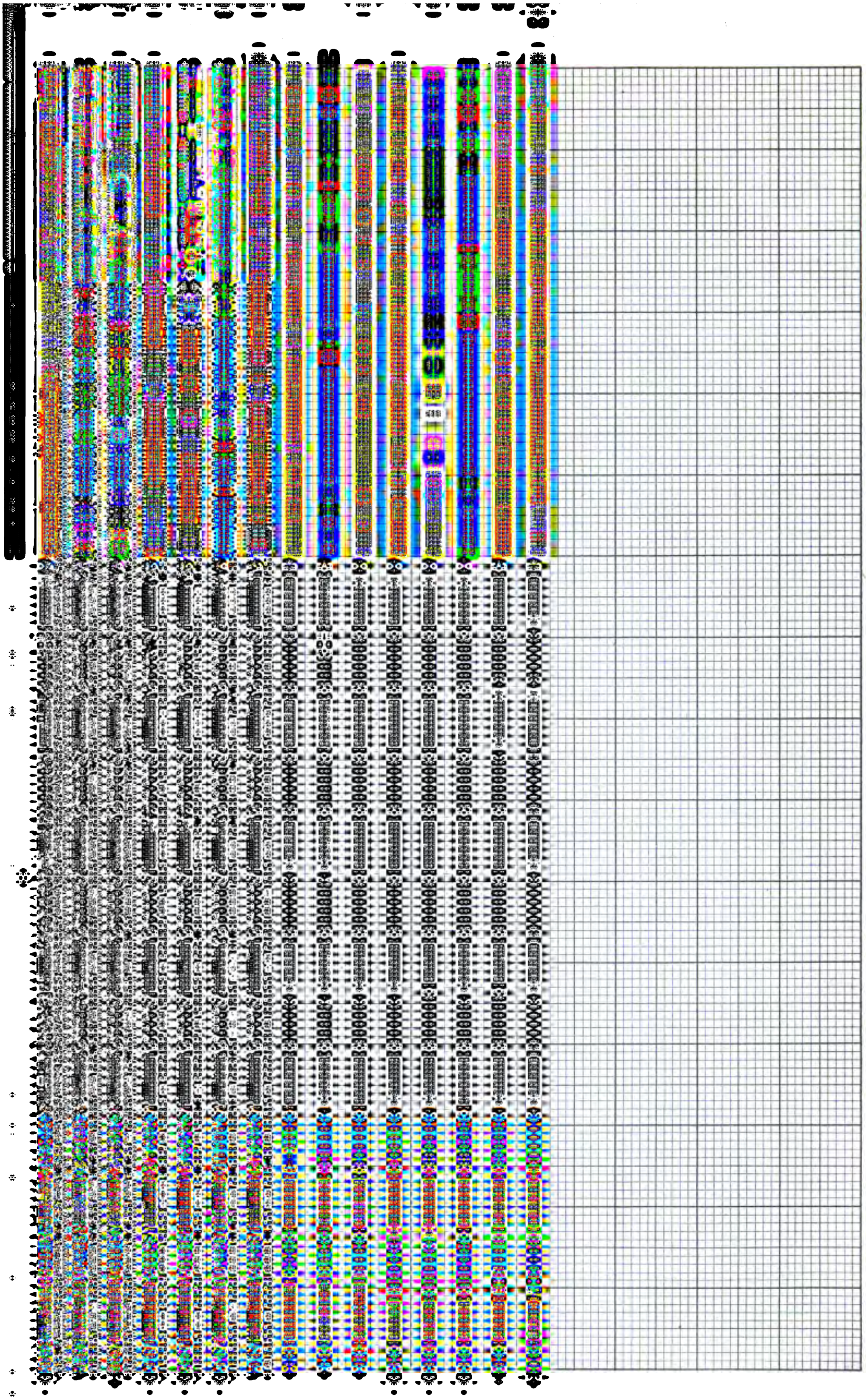


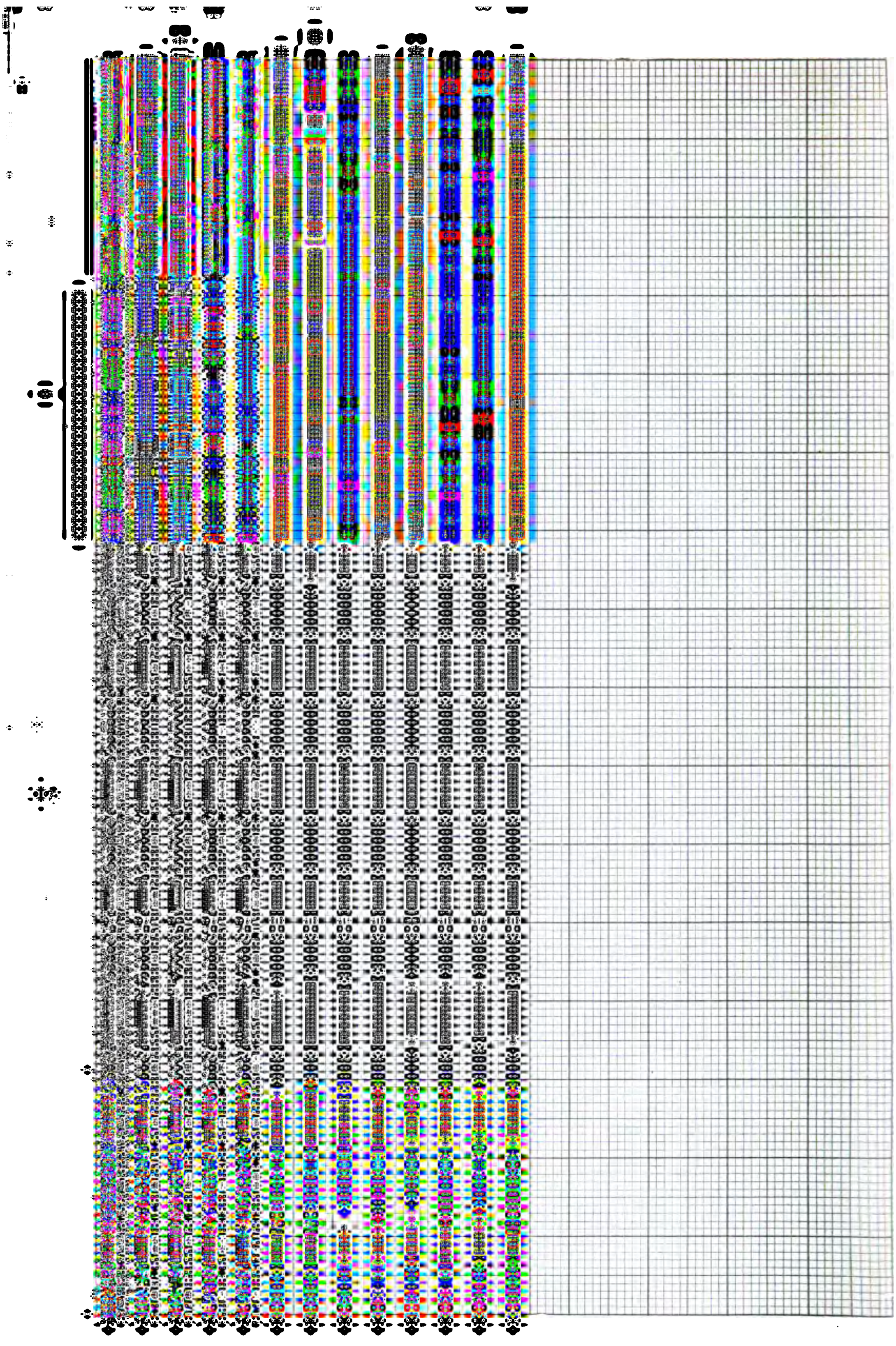


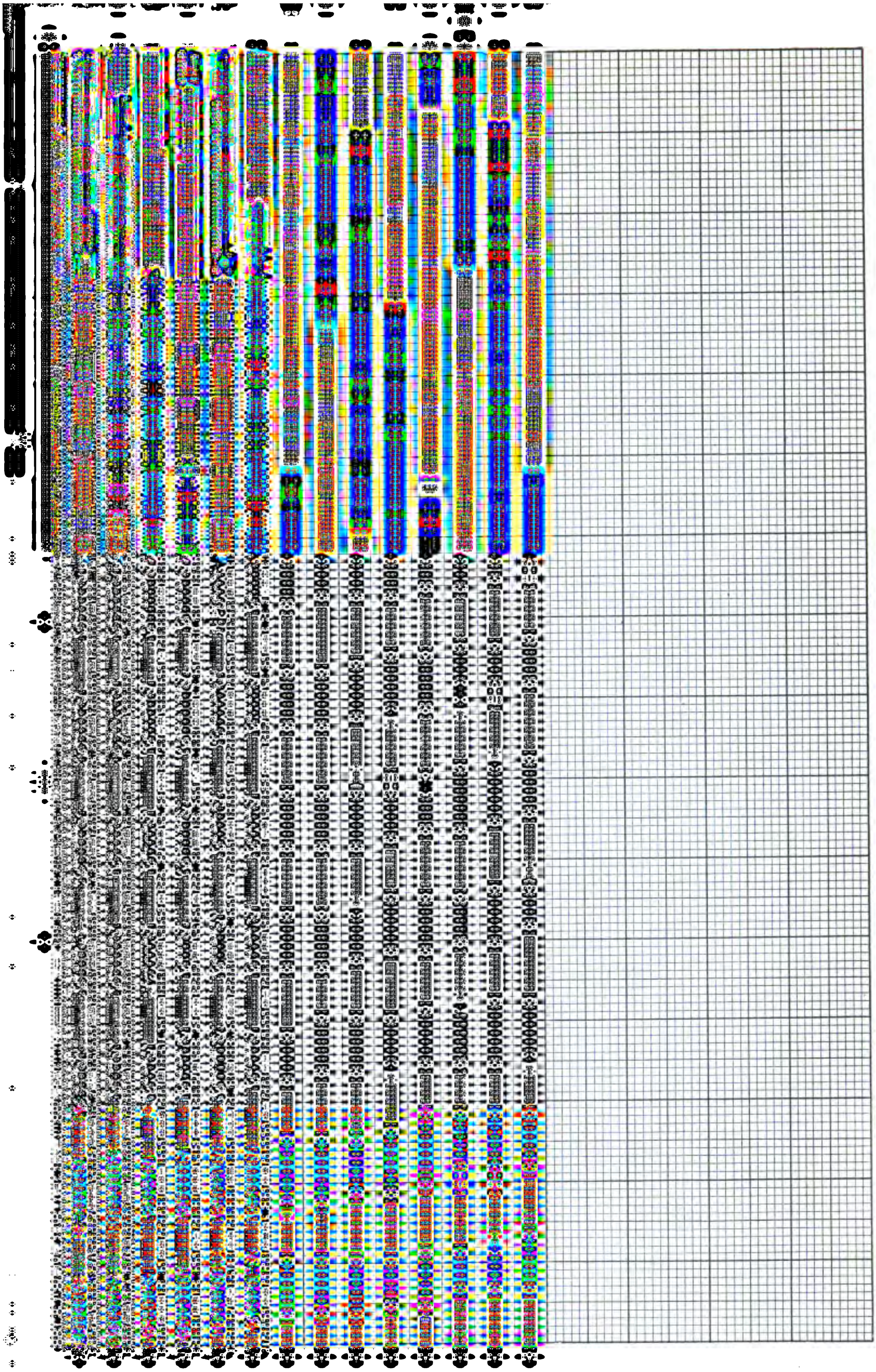


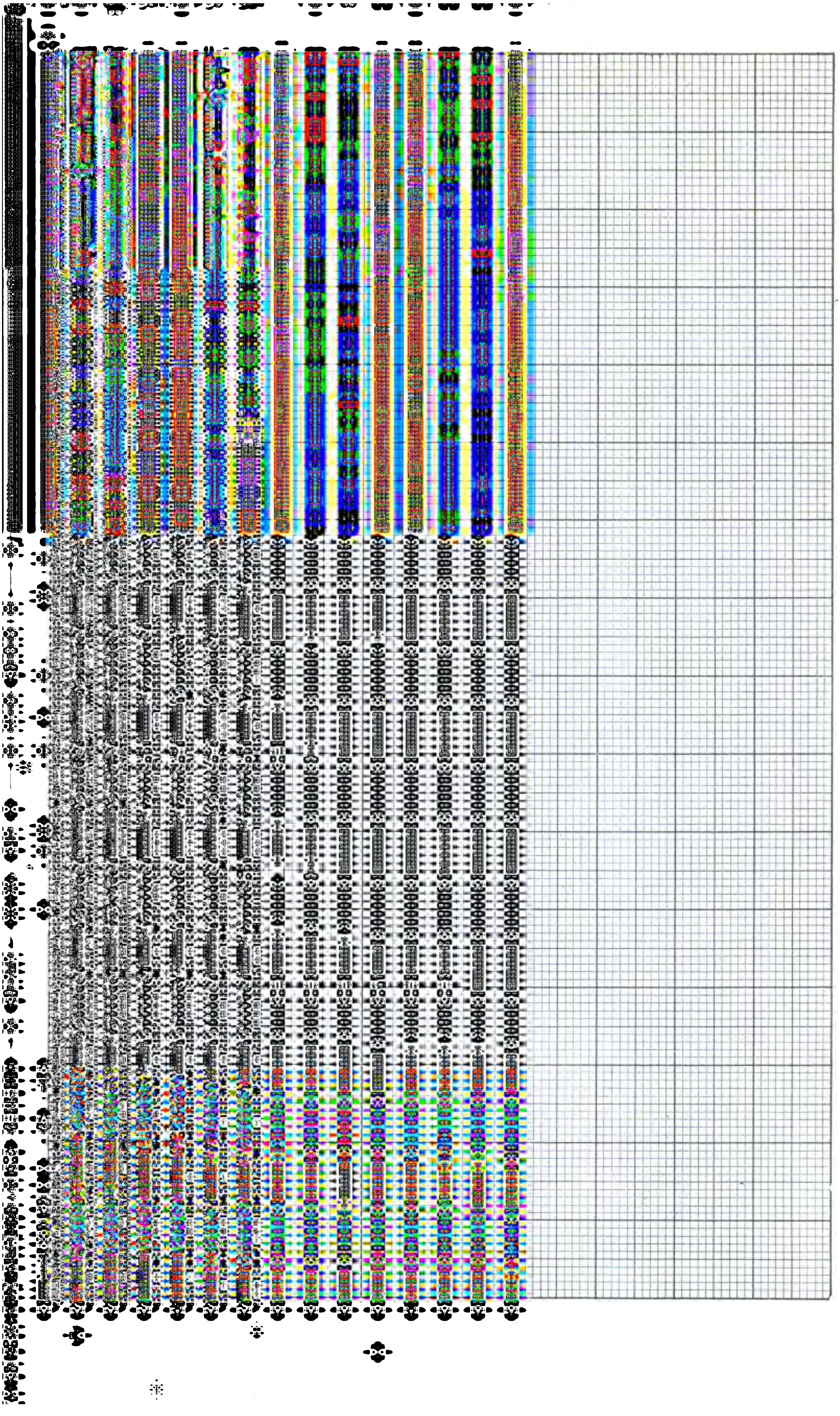


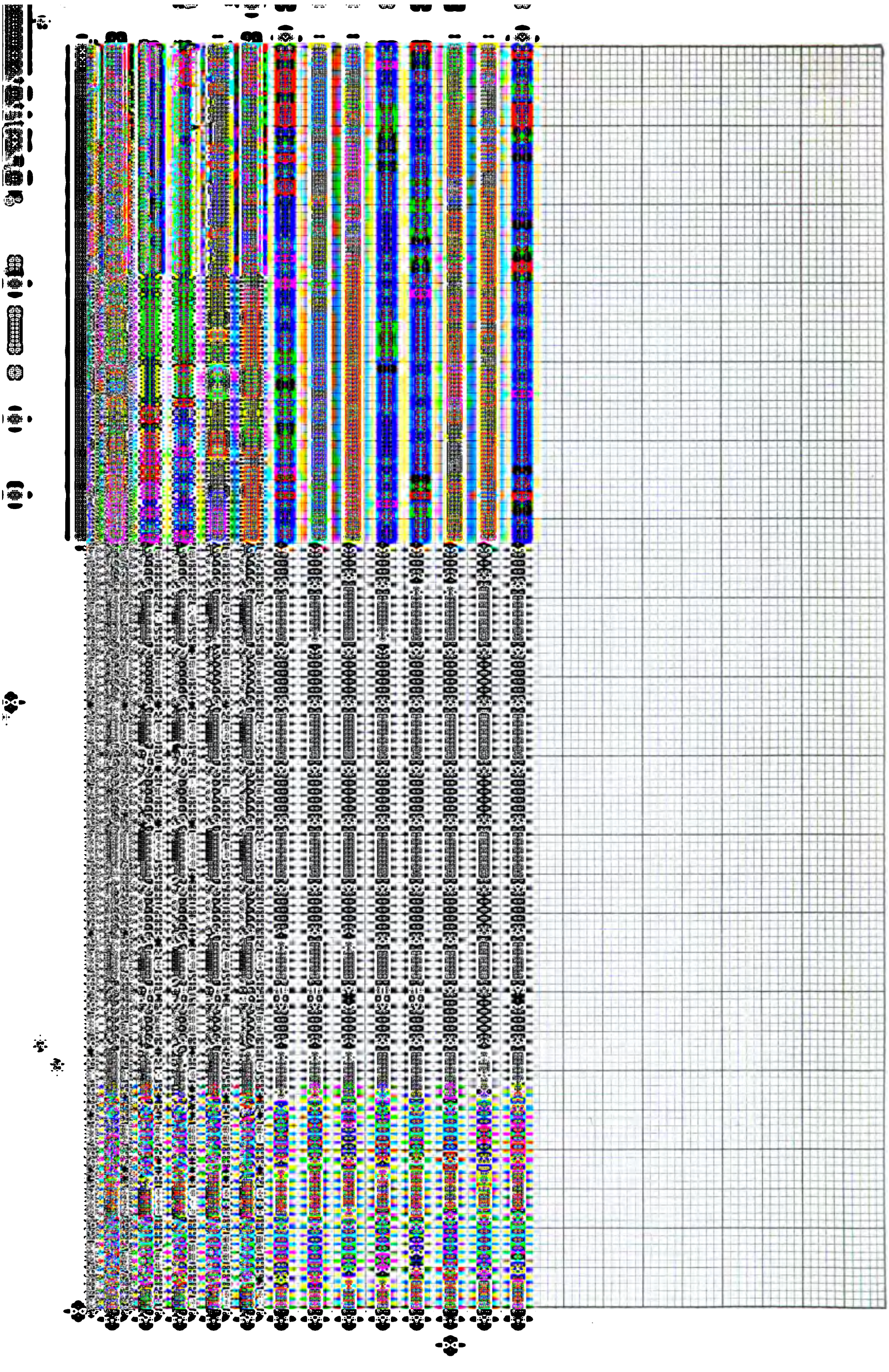


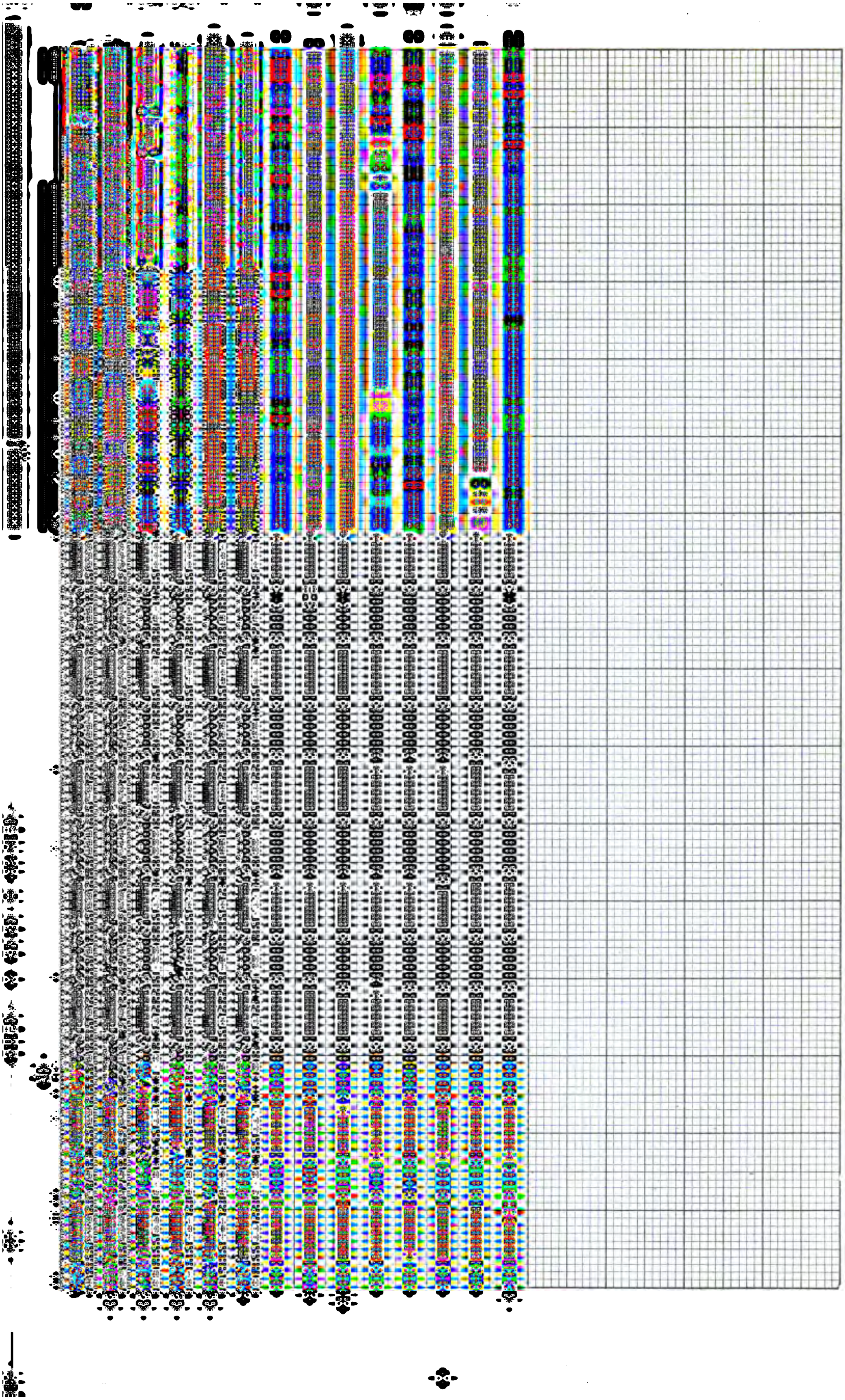


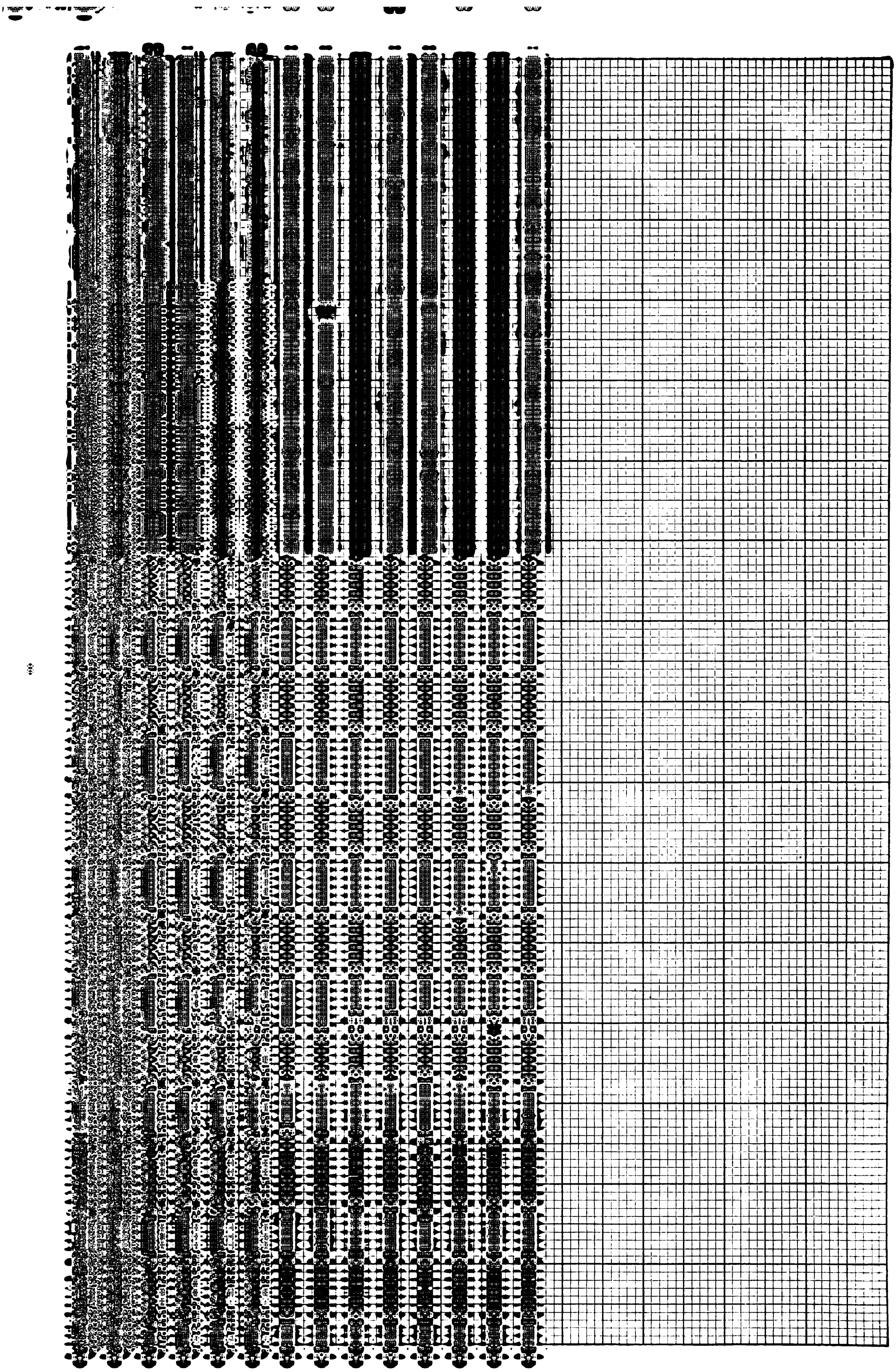


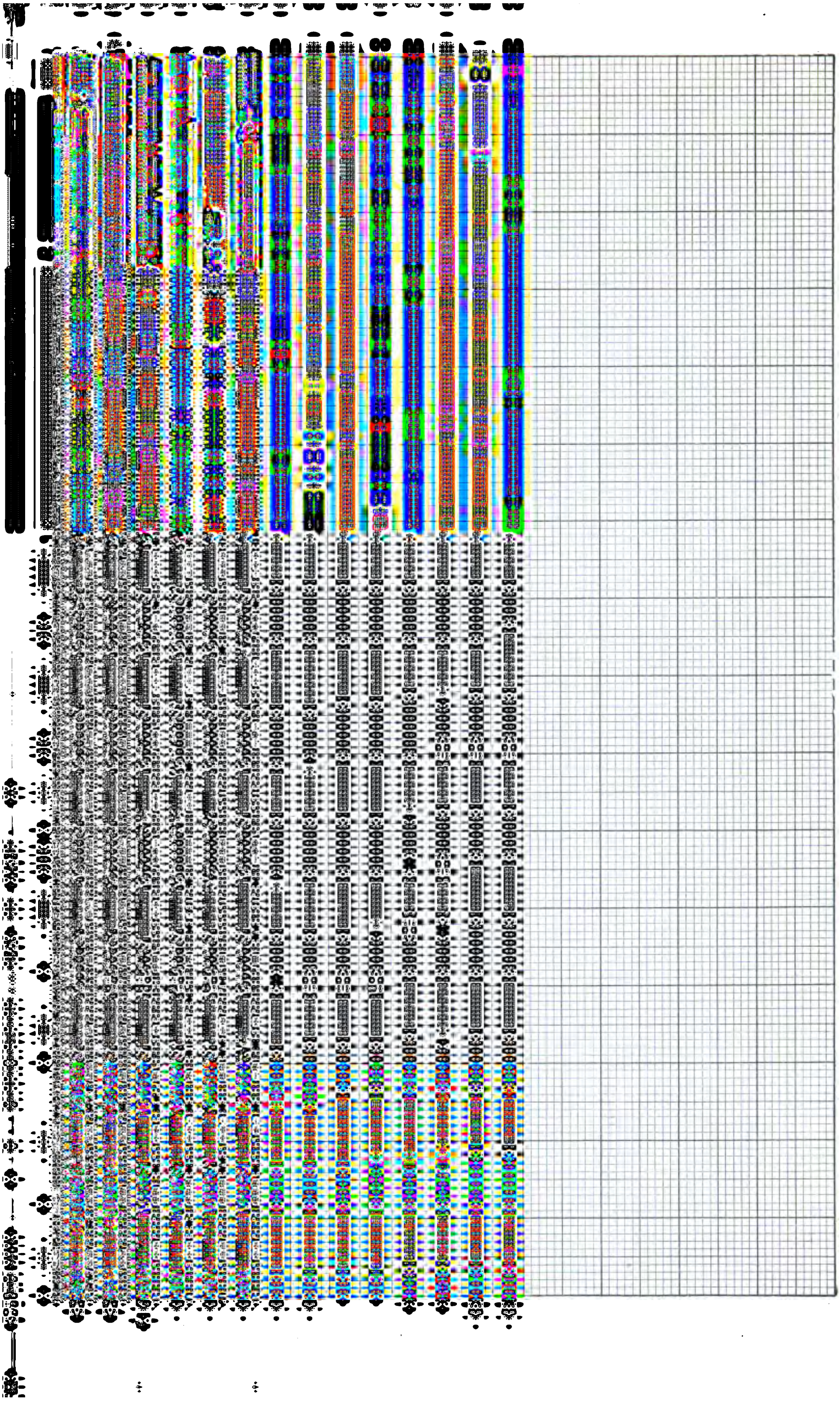












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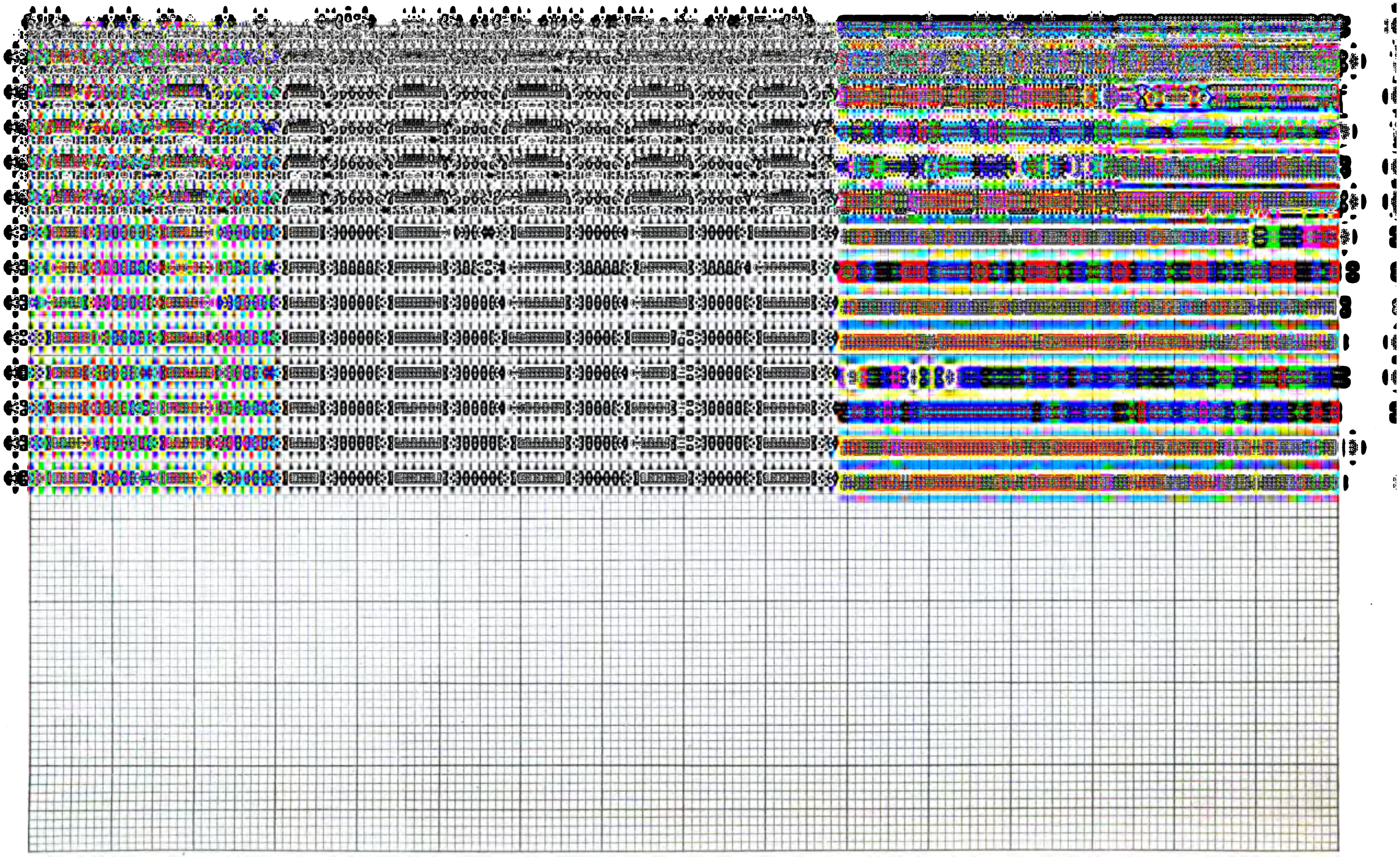

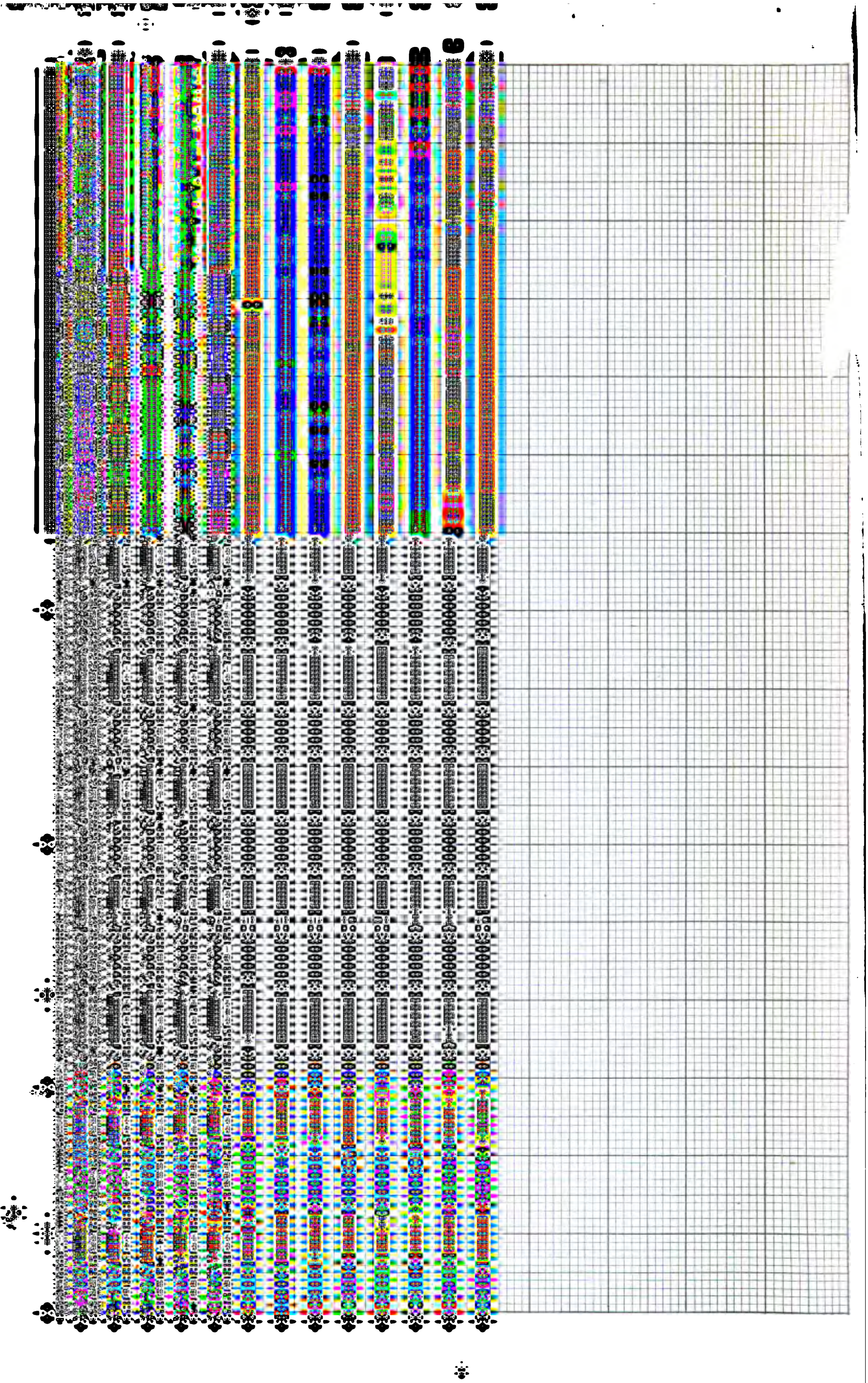

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